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High energy ion beam irradiation of polymers for electronic applications

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Abstract

Ion irradiation of polymers offers a number of interesting possibilities for applications. In the case of latent tracks, radiochemical changes, phase transitions, alterations of the intrinsic free volume, or radiation induced defects can be exploited – the latter ones to trap mobile impurities. These approaches are useful to form, e.g. new types of sensors.

Apart from this, etched tracks in polymers offer a vast range of possibilities. Practically any material – including colloides and nanocrystals – can be inserted into these pores to form nanowires or nanotubules. Sequential deposition can be made as well in radial as axial direction to form complex nanostructures. Combination with lithography enables one to form different types of novel transistors, microcapacitors, -magnets, -transformers and -sensors. Also sterilizing foils for medicine and packing industry have been made in this way. A number of new ideas are presented how to proceed further in this field.

Keywords: Latent ion tracks; Etched ion tracks; Polymers; Applications; Electronics

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1. Introduction

The recent years have brought a renaissance of interest in ion tracks in polymers, for the sake of novel applications. One has to distinguish between

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the application of latent, i.e. as-implanted ion tracks, and etched tracks. The primary deposition of high energy (∼MeV to ∼GeV) inside a tiny target volume ('ion track core', $\sim 10^{-15\ldots-14}$ cm^3), and within an extremely short time ($\sim 10^{-17\ldots-15}$ s), by swift heavy ions leads to dramatic transient modifications of the materials via chemical and structural changes (e.g. the transient breaking of all bonds), with accompanying heat and pressure pulses. In spite of repair of much of the primary damage during the annealing phase after the ion impact ('thermal spike', $\sim 10^{-12\ldots-11}$ s), quite a number of irreversible changes remain. The cylindrical zones with altered properties where these changes prevail are the so-called "*latent tracks*". Dissolution of the latent track material by suitable agents ('etching') leads to the formation of pores, the so-called "*etched tracks*". By careful selection of projectile, target, etchant, and etching conditions, the etched track can be tailored towards any required shape, such as cylindrical, conical, or hyperbolic, transmittent (in thin foils) or non-transmittent.

In a recent paper [1] there have been outlined the newly emerging possibilities, and the strategies were summarized that had been initiated at that time. Only a few applications that are based on *latent* tracks have emerged since then. For contrast, *etched* tracks in polymers appear to have a much greater application potential. Compact rods and tubules as well as dispersed nanosized matter can be embedded within the etched tracks, to form the base of various applications. Though the number of present applications is still quite limited, it appears that ion tracks in polymer foils will soon find a multitude of new interesting applications not only in electronics but also in other fields such as medicine or optics. Some of them are summarized here, and a few examples are described in detail.

2. Applications of latent tracks

Latent tracks in polymers are characterized by (1) structural changes such as an altered free volume, carbonaceous clusters, etc., (2) a high density of radicals, (3) chemical changes such as unsaturations, double bonds, and (4) phase transformations, such as amorphisation. These changes are responsible for the four major strategies that have emerged for latent track applications: (1) exploitation of the modified transport properties along ion tracks, (2) trapping of mobile (e.g. metallic) atoms, molecules or clusters along the tracks, (3) exploitation of the material's chemical changes, and (4) making use of ion-induced phase transitions.

The first concept is exploited in using swift heavy ion irradiated polymer foils as seals to protect sensitive volumes against penetration of ambient dust and moisture while maintaining pressure equilibrium and gas exchange with the ambient. It can also be realized, together with the second concept, by the decoration of latent tracks by liquids and solutes dissolved in them [2], by grafting specific monomers onto the host polymer along the ion track [3]; or by trapping of metal atoms along the tracks that will thereafter cluster to form conducting nanowires. It has also been found that metallic nanoclusters in metal/polymer composites can be aligned along the tracks towards pearls-on-a-string-like patterns [4].

The third concept has been realized already manyfold, e.g. by producing dangling bonds along the tracks that enable protonic transport for hydrogen sensing purpose [5], by the radiochemical destruction of sensitive polymers such as polysilanes that form SiC rods [6–11], or by organometals that form precipitates of metals or metal oxides, sulfides etc., along the tracks upon ion irradiation. The emerging anisotropy of the conductivity of such irradiated materials [12] can be used to create novel electronic structures (the so-called TEAMS (= tunable electronic anisotropic materials on semiconductors) structures [13]) for advanced devices. The last concept, finally-phase transformations-hardly applies to irradiated polymers. Here, rather carbon allotropes are used, such as diamond [14], or fullerite [6] in which conducting latent tracks are formed upon irradiation, by transformation of sp3 bonds towards sp2 along the ion paths.

Summarizing, most of these concepts aim at producing conducting nanowires in one or the other way. In contact with semiconducting substrates these nanowires form Schottky nanodiodes. The multitude of such parallel nanodiodes, eventually interacting with each other, enables one to construct electronic building blocks of higher complexity, which we denoted as TEMIPOS (tunable electronic material with irradiated polymers on semiconductor) structures [13]. One could also think at producing arrays of nanoscopic thermocouples by appropriate material's choice for both the nanowires and substrate, for future IR sensor chips. Most of these technological approaches are still in an embryonic state.

3. Preparation of etched tracks for applications

Etched tracks can be filled, in principle, with any material, and the embedded matter can be arranged as either massive wires (also called: "fibers, fibrilles") or tubules, or it just can be dispersed discontinuously as small nanoparticles along the track length. The techniques how to accomplish this – such as galvanic deposition, chemical deposition, pressure injection, grafting, in-situ polymerization, evaporation, etc. – have been developed in detail during the past decade [8,15–29]. For some of them, e.g. the chemical deposition techniques, it is important to provide a sufficient areal density of nucleation centers on the track walls to obtain a sufficiently dense coverage of the required material. Such nucleation centers can be produced chemically, or radiochemically by irradiation with laser beams or energetic ions. The latter approach provides a tool to tailor the track structures axially via the ranges of the used ions (Fig. 1) [1,30]. Sequential galvanic deposition of different materials leads to axially structured elements, and sequential chemical deposition leads to radially structured elements.

Of course, some nucleation centers are always present on the track walls, which are given by the intrinsic polymeric surface defects such as e.g. radicals or impurity atoms, but their areal density is rather low for the modern synthetic polymers. If, e.g. during a chemical deposition process, one restricts to these intrinsic nucleation centers only, one will obtain only a few dispersed (semi)conducting nanocrystals on the ion track walls (Fig. 1). In fact, this may even be beneficial for tailoring the track resistivity an capacity towards any desired value, via the choice of the chemical deposition time that determines the size of the precipitating nanoclusters, and hence the intercluster distance–consequently also the type of the electrical conduction mechanism, and its dependence on voltage and temperature. Apart from the possibility to tailor the electrical properties of ion tracks by the precipitation of nanoclusters, the latter ones also possess other peculiar electronic and optical properties such as a high charge storage capacity and fluorescence, etc., which may lead to new yet unknown challenging ion track applications.

Another possibility to cover the track walls with nanoclusters is to introduce a colloidal solution into the etched tracks. Some colloidal solutions are already commercially available,[1] others have to be made in the lab (e.g. TiO_2 or $LiNbO_3$ [1]). When working with such colloidal solutions one should always take into account that one does not work with the nanoparticle material only, but that the particles are surrounded by organic ligands for stabilization, and that commercial colloidal solution may still contain other – partly unknown – additives. These materials might eventually modify the nanoparticle properties in an unpredictable way.

4. Applications of etched tracks

4.1. Overview

Many advanced applications of etched tracks have already been reported or at least discussed, such as the formation of nanosized or microsized diodes [27,31,32]-eventually even light-emitting ones [33,34]-, tunnelling structures [27,35], field effect transistors [36–39], devices to control the permselectivity of electrolytes [40], temperature-sensitive valves [41], miniaturized magnetic field sensors [42,43], electrolytic oxygen sensors

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[1] E.g. SiO_2, offered by Baier Ltd. under the trade name 'Levasil', or colloidal gold, offered by Sigma-Aldrich, or, for etched tracks in the μm size, also conducting silver paste.

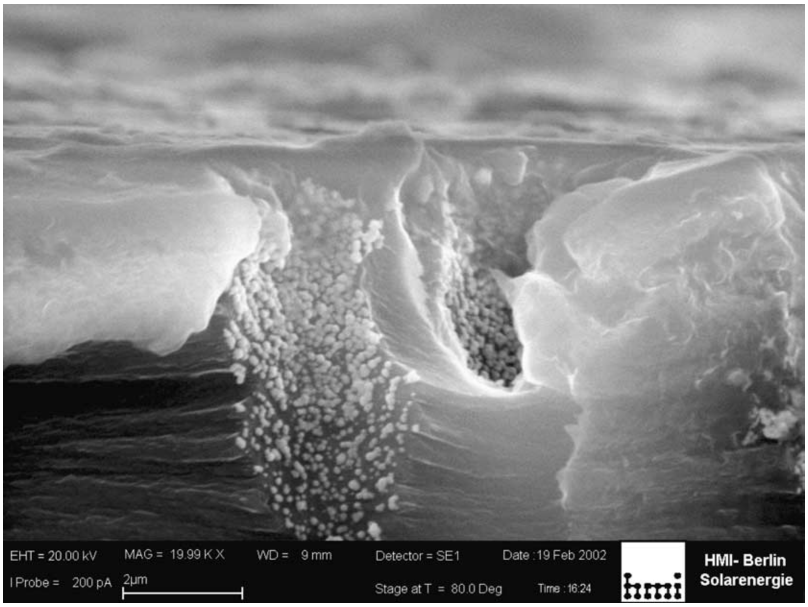

Fig. 1. Scanning microscopic view of a crack through a conical etched track in polyimide, upon which silver was chemically deposited. As the sample had been irradiated with low energy ions (50 keV Ar^+) before the silver deposition, the surface region was highly enriched with nucleation centers, hence a continuous Ag film is deposited. On the other hand, inside the etched tracks, Ag precipitates only on the very few intrinsic polymeric defects.

[44,45], in vivo long-time storage vessels for drugs [46], miniaturized Li batteries [47], and sensors for temperature [48], pressure [48,49], humidity [1,30,48], and ammonia [50], photoreceptor arrays as the first stage of artificial eyes [51], enzymatic bioreactors [52], preparative work for MAIA (multianalyte immuno array) antibody chips [53], sterilizing foils as plasters in medicine and as anti-ageing packing materials for food and flowers, based on nanofibrilles or nanoparticles [51,54] of the TiO_2 phase anatase, and microwave filters [55]. Some of these applications need several working steps such as the sequential galvanic deposition of differently semiconducting (i.e. n- and p-conducting) materials, or of metallic and semiconducting wires (e.g. Cu and Se, or Ni and CdSe/CdTe) to give rise to nanometric diodes. Sequential axial (e.g. galvanic) deposition of different ferromagnetic materials may lead to giant magnetic resonance (GMR) devices [56]. Sequential radial (e.g. chemical) deposition of different semiconductors, or of metals and semiconductors leads to the formation of concentric diodes, and the sequential radial deposition metal/insulator/metal structures leads to concentric nanocapacitors. Connecting metallic wires embedded in etched tracks with suitable metal contact stripes evaporated onto the surface of microporous polymer foils leads to the formation of microinductances and microtransformers [1,18,30].

4.2. Ion track-based microcapacitors

Our first experience with nanocapacitor formation (Fig. 2(a)) was ambiguous. Though the approach is, in principle, feasible, we have often experienced local shortcuts between the two capacitor electrodes in some tracks, so that their equivalent circuits were always described by a (occasionally rather small) resistance in parallel with the capacitor. A much easier approach is to put a grid of opposing metal-filled ion tracks, which are arranged like "classical" capacitor plates, onto two different potentials, see Fig. 2(b). Such simple arrangements (Fig. 2(c)) showed a nearly constant capacity up to at least 1 GHz. In fact, the replacement of the "classical" capacitor plates (Fig. 2(c)) by ion track-made grids (Fig.

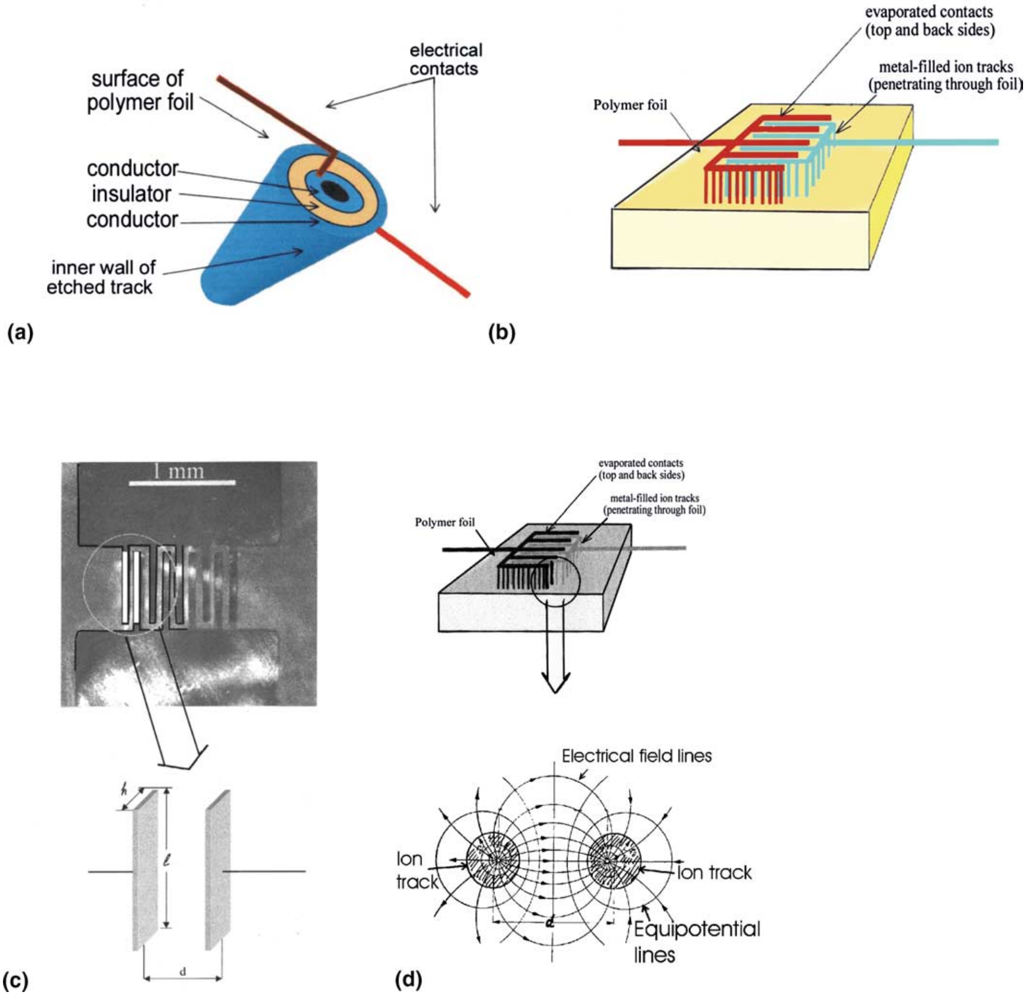

Fig. 2. Principle sketch of (a) an ion track-based nanocapacitor made by sequential concentrical deposition of three layers, and (b) of an ion-track based microcapacitor, made by combination of selected (via lithography) conducting ion tracks and evaporated contacts. (c) Image of the first microcapacitor prototype, with illustration of the capacitor function of that device. (d) Image of the microcapacitor fine structure made of conducting tracks, with illustration of the radial electric field around such opposing tracks.

2(d)) increases the capacity considerably, as can easily be derived from the corresponding well-known text book formulae [57]. Polyimide is a very suitable polymer for that purpose, as its dielectricity coefficient is only marginally frequency dependent, even at the highest frequencies [30].

4.3. Ion track-based micromagnets

We reported in [1] and [18] on the first prototypes of ion-track based micromagnets and microtransformers. They showed good operation up to ~1/2 GHz, with quality factors approaching up to ~10. These microtransformers can suffer an astonishing electrical and thermal load, due to the heat resistant carrier polymer (Kapton) used, and the rather thick copper tubules employed at that occasion, Fig. 3(a) and (b). We have intentionally designed these magnets and transformers with metallic tubules instead of metallic wires, in order to transmit through them in future cooling gases or liquids, to enable even higher thermal loads.

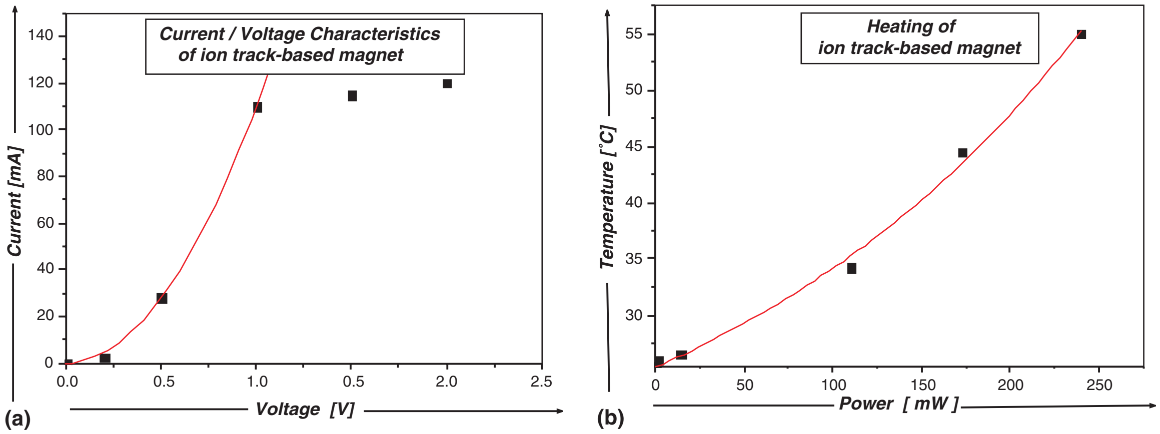

Fig. 3. (a) Current/voltage relation of a micromagnet. The quadratic I/V correlation of that magnet ended abruptly at ~1 V, when the interface between the conducting ion track and the evaporated surface contact failed thermally. After a near-constant current at increasing voltage, the critical contact point burnt completely at ~2 V. (b) Temperature rise of a micromagnet in dependence of the deposited power.

In fact, the first prototypes survived already some 250 mW. Failure occurred at the contact points between nanotubules and evaporated contacts which hence have to be improved.

By combination of lithography with ion track technology, K. Hjort and his group succeeded meanwhile to obtain much smaller magnets with better quality factors, capable for operation up to the ~50 GHz range [58]. Future micromagnets should be made of ultrapure aluminium instead of copper, to reduce their resistance even more. We hope to be able to produce in this way micro-transformers that are competitive for space applications in miniaturized satellites.[2]

4.4. Ion track-based polymer/silicon hybride structures

Recently we developed a new electronic device which is, loosely spoken, a MOS-FET-like structure, with its dielectric layer "shortcut" by high Ohmic conducting tracks. The latter ones were prepared by deposition of Ag nanoclusters similarly as shown in Fig. 1. This structure that we denoted as "TEMPOS" (= tunable electronic material with pores in oxide on silicon) [59–61] exhibits a number of specific properties which make it stand in between tunable resistors, capacitors, diodes, transistors, and sensors. It is worth mentioning that this structure shows inherent current instabilities in certain working points that make it behave similar to Esaki diodes or unijunction transistors in these cases. These structures are highly interesting as they enable, in principle, due to their negative resistances, the transistor-less (hence more compact) construction of computers.

In the frame of ion irradiation of polymers there arose the question whether the dielectric material of these TEMPOS structures (usually silicon oxide or silicon oxynitride) could not be replaced by other, polymeric matter. This would enable one to combine the great number of advantages of polymers in electronics with the well-established silicon technology, and these silicon/polymer/track hybride structures would make the transition from the contemporary silicon technology to a future polymeric technology more acceptable for the electronic industry.

In fact, our first attempts to use the photoresist AZ1350 as the dielectric layer on silicon were successful. A ~300 nm thick photoresist layer was spin-coated on a n-Si wafer, and subsequently irradiated with 300 MeV Au^{26+} ions at fluences of ~10^7 cm^{-2}. According to the company's recipe, the sample was then subject to the corresponding

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[2] A. Demyanov, pers. commun. 2002.

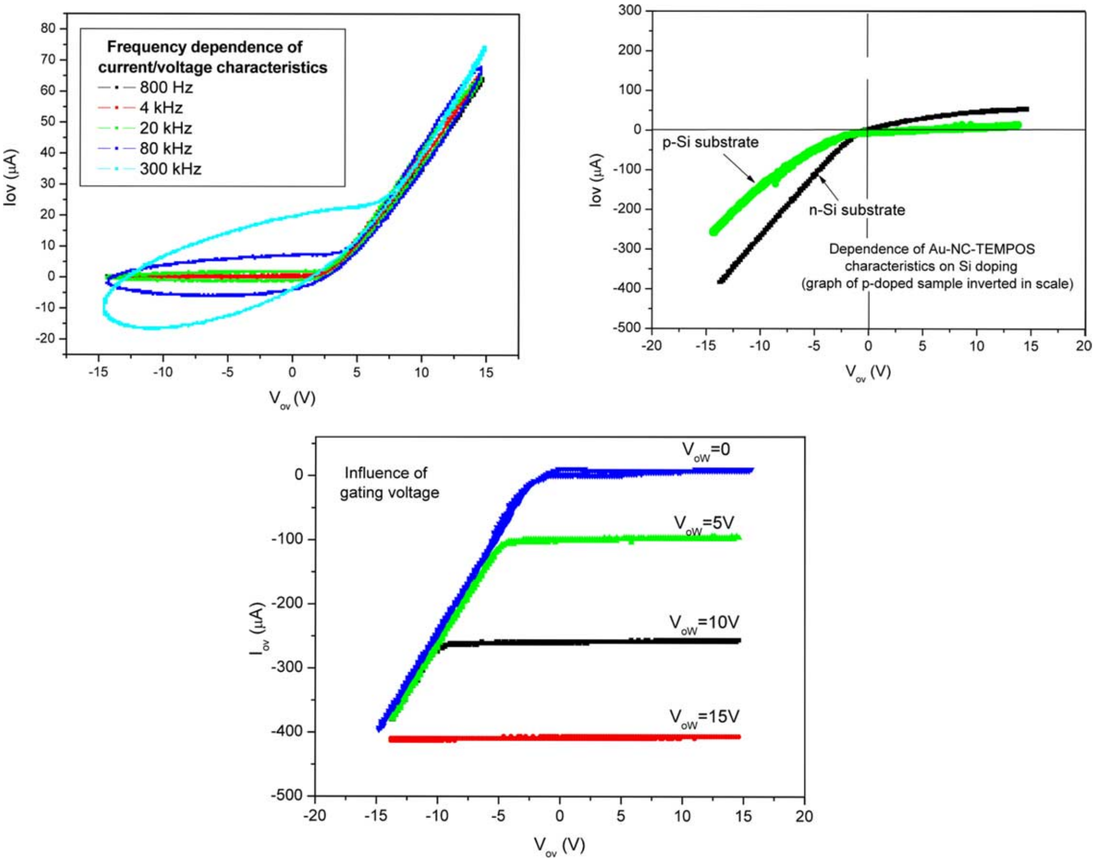

Fig. 4. Electrical characterisation of TEMIPOS structures with Au nanoclusters. V_{ov} is the voltage applied from top (microporous polymer layer; contact ***o***) to bottom (Si substrate; contact ***v***) of the TEMIPOS structure; I_{ov} is the current measured along this path. The gating voltage V_{ow} is applied between the top contact ***o*** and another top contact ***w***.

developer for ~1 min at ambient temperature, and then rinsed with water and dried. After depositing a droplet of distilled water onto that structure and contacting the latter with a tiny needle, an electric current could be measured from the droplet to the silicon substrate, thus indicating the full track opening. Subsequently a droplet of a 10% diluted commercial colloidal gold solution was allowed to penetrate into the structure, and to dry.

The contacted structure reveals electronic properties similarly to those ones prepared earlier, Fig. 4. We called it "TEMIPOS", see above. The TEMIPOS structures obtained thus far behave like tunable diodes. Under some working conditions they also show current instabilities (Fig. 5), as do the corresponding TEMPOS structures with SiO_2 or SiON dielectrica.

5. Summary

Ion tracks, specifically etched tracks, alone or in combination with lithography, enable many possibilities for creating novel deep micro-and nanostructures within polymer foils that are difficult to produce or even inaccessible by other techniques. First prototypes of a number of ion track-based electronic elements such as resistors, diodes, capacitors, magnets, transformers, transistors, and several types of sensors have been created, and the first hybride track-based silicon/

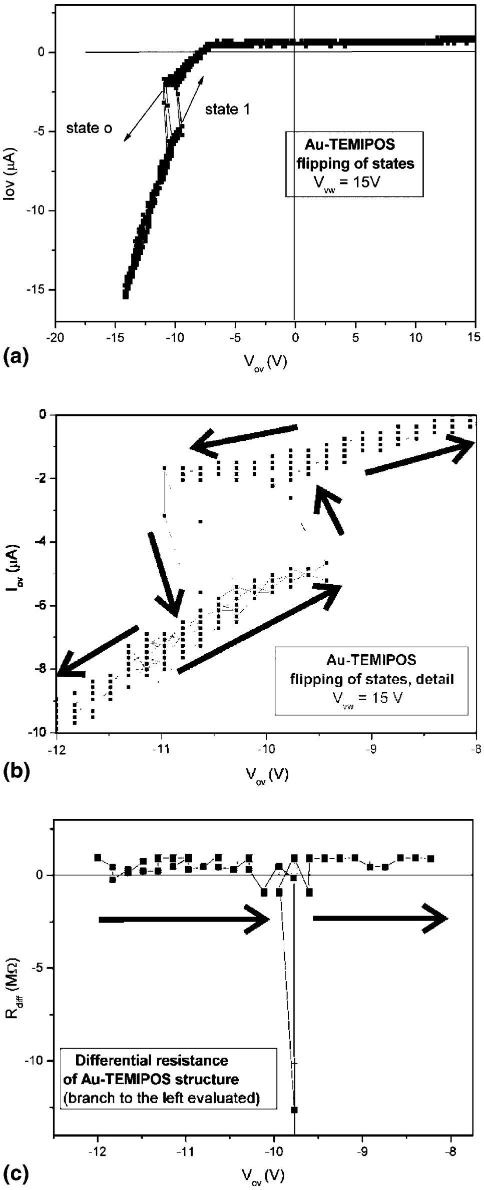

Fig. 5. An example for the current instabilites found for TEMIPOS structures. (a) Overall view of the current/voltage characteristics (150 Hz AC applied; zero gating voltage). (b) Detailed view of the instability region (10 cycles depicted); where reproducible jumps between a state "0" and a state "1" are observed. (c) The local differentiation of (b) reveals that the resistance in the instability region approaches to zero or even becomes negative, thus indicating the potential for applications in active electronic devices.

polymer device has been tested successfully. It will be, however, still a long way up to the industrial application of many of the topics touched here briefly.

Finally it should be mentioned that self-ordering thin porous ceramic foils, especially of alumina, have emerged as serious competitors for microporous polymer foils in many fields, due to their higher temperature stability, higher rigidity and more regular pore arrangement. However, their brittleness, higher price and smaller size restrict their applicability, so that the application potential of ion-track based microporous foils is nevertheless still tremendous.

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