

Early Attainments of Porous Silicon Carbide Technology: a Bibliographic Digest

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Abstract. This work presents a bibliographic review on a promising functional material - porous silicon carbide (PSC). The work reviews selected sources, which describe the main achievements that formed the technological basis for PSC yet in the first decade of the 2000s, but were often ignored in the later research and publications. It is expected that this selection would be useful for specialists in semiconductor physics, engineers, and technologists working in this field.

1. INTRODUCTION

The first semiconductor material ever made porous was silicon (PS). The possibility of the formation of porous structures in the bulk of monocrystalline silicon was discovered in 1956 by A. Uhlir, Jr. during a study of the process of electrochemical polishing of the surface of silicon wafers in solutions of hydrofluoric acid (HF) at Bell Laboratories (USA) [1]. For a long time after that, PS was considered only as a laboratory curiosity. The first practical use of PS was demonstrated in 1969 by Sony, which used it to fabricate isolation regions in microcircuits. In the mid-seventies, several groups continued their efforts to obtain various structures “silicon-on-insulator”, which, however, did not lead to the development of serial industrial devices; for review, see, e.g., [2]. A new explosive interest in PS was sparked by the discovery in 1990 by L.T. Canhan (Royal Signals and Radar Establishment, UK) of PS emission of visible light. The PS appeared to emit light at the room temperature in the red-orange spectral range, when excited by a blue or green laser [3]. The author explained the nature of the observed photoluminescence in PS by the emergence of the effect of size quantization, which lead to the emission of photons with energy that significantly exceeded the band gap of bulk crystalline Si. However,

to this day, the interpretation of this phenomenon is the subject of scientific debate. At the same time, the origins of many other properties of PS remain unclear. For example, there is no unified theory of PS formation and processes of charge carrier transfer in PS, nor there is a common understanding of the reasons for the high resistivity of a porous material. Here we note that Lee Canham subsequently pioneered the biomedical applications of porous silicon as well [4]. To date, there have been several generalizing reviews on the recent results of obtaining porous structures based on semiconductor materials and compounds and the prospects for their application for modern micro- and nanoelectronics, sensing and biotechnologies [5-11].

Interest in silicon carbide (SiC) as a semiconductor material started in the 1950s. It was known that from the point of view of practical use, this material had fundamental physical properties superior to many of those of silicon (Si), which at that time served as the main material of semiconductor electronics. The wide band gap in a broad energy range (from 2.4 to 3.3 eV for various practical polytypes of SiC), high thermal conductivity, high electric breakdown field, radiation resistance in combination with a high melting point made SiC a material of choice for developing new promising electronic

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devices for power distribution systems, high-temperature gas turbine engines, high-frequency power devices and detectors able to work effectively in high-temperature or corrosive environments. As predicted by William B. Shockley (*American physicist, semiconductor researcher, Nobel Prize winner in physics in 1956*) in the early fifties such a material (SiC) will quickly “replace Si”. However, despite the recognized potential, difficulties in growing high-quality, large-size single crystals have hampered electronic applications of SiC and lead to the stagnation in early research. The technology for the production of high-quality bulk SiC single crystals became successful only after 1990 using the method of sublimation with a seed (modified Lely method, LETI method) [12]. Plates of 4H- and 6H-SiC polytypes with a diameter of 2–3 inches obtained from the bulk material became commercially available. With these successes, a breakthrough in the technology of epitaxial growth of SiC using the chemical vapor deposition (CVD) method [13–15] took place. This has significantly accelerated the progress in the development of electronic devices based on SiC as well as in the use of porous SiC in a number of promising new applications.

In particular, a six-year program (DURINT) with a multi-million dollar funding was launched in the US in 2001. This program was launched with the aim of developing a porous SiC technology in order to study the possibilities of its use as a material for catalysis and biotechnology, and also as substrates that would allow for improving the quality of epitaxy of SiC and gallium nitride (GaN). The main achievements of the international group of researchers involved in this activity are summarized in the book [16]. This publication has accumulated the research results of the period 2001–2006 carried out within the framework of the program “Nanoporous Templates for Large Defect Reduction in SiC and GaN, Nanocatalysis, Magnetic Clusters, and Biotechnology”. These studies, in particular, concerned such topics as methods of obtaining porous structures, epitaxial growth of SiC and GaN on porous SiC and the first achievements in the field of using porous SiC for catalytic and biotechnological applications.

2. POROUS SiC FORMATION

Porous structures based on semiconductors are usually obtained by electrochemical anodization of plates of the starting single-crystal material in electrolytes based on water-alcohol mixtures of hydrofluoric acid (HF: H₂O: C₂H₅OH) in various ratios. For anodization, two or three electrode electrochemical cells are used, which are connected to a power source that regulates the current and voltage of the electrical circuit. The plates act as an anode, while platinum electrodes are used as a

cathode and an additional counter electrode. The electrochemistry of semiconductor materials as a branch of physical chemistry began in the 1950s. Up to the present time, many models have been proposed to describe the process of formation of porous structures. These models can be conditionally divided into two groups. The first one includes electrochemical etching models that describe electrochemical reactions between an electrolyte and a semiconductor. Models forming the second group take into account the contribution of physical processes, in particular, diffusion charge transfer. The first attempts to explain the formations of a porous structure were undertaken for PS (see the review by R.L. Smith and S.D. Collins [17]). However, later experiments on the preparation of porous materials based on AIIBV and AIIBVI semiconductors showed that the models developed for Si cannot fully describe the formation of porous semiconductors, thus requiring new approaches [18]. Without going into details, it can be noted that the analysis of the current state of affairs shows that the problem of pore formation during anodization of semiconductor materials is still under discussion, and the existing theories of pore formation are diverse, complex and often contradictory. In addition, along with technological progress, it becomes reasonable to expand the parameters of the accepted “classical” classification of porous structures. Until now, in PS technology as well as in that of other porous semiconductors the classification of the porous structures by pore size is used in accordance with the recommendations of the IUPAC (International Union of Pure and Applied Chemistry) [19]. Following these recommendations, all possible types of structures are divided into three types: macroporous (pore diameter $d > 50$ nm), mesoporous ($2 < d < 50$ nm), and nanoporous ($d < 2$ nm). Recently, it has been suggested that from the point of view of modern applications, an additional integral parameter such as the total specific internal surface area of porous structures may be considered as an important characteristic [20].

Porous SiC (PSC) was first reported in 1993 by Joseph Shor and his colleagues from Kulite Semiconductor Products and Columbia University, who actively studied electrochemical treatment of SiC [21, 22]. The authors reported on the production of PSC by anodization of 6H-SiC with *n*-type conductivity in a three-electrode Teflon electrochemical cell using a 2.5% aqueous solution of HF as an electrolyte [23]. Transmission electron microscopy (TEM) data showed that pores with a diameter of 10 to 30 nm propagated within the bulk of the anodized material in different crystallographic directions. A suggestion was made that the formed pores are located at the sites of defects in the initial samples, which were supposed to act as centers of local electrochemical

etching. First report on the study of optical properties of porous structures based on 6H-SiC with *p*-type conductivity was published by Shor et al. in 1994 [24, 25].

One important property of PSC, namely, its high susceptibility to oxidation, was also demonstrated and used by Shor et al. [26]. The authors of Ref. [26] suggested that the large surface area and relatively small interpore spacing of porous semiconductors should make them more susceptible to oxidation than normal bulk material. This suggestion was confirmed by a successful attempt towards patterning of SiC in the form of commercially available wafers. Anodization of 6H-SiC through a sputtered Cr/Au mask was held in a standard Teflon cell under ultraviolet (UV) illumination. After anodization the porous layers were subjected to wet oxidation. The oxidized porous SiC was successfully removed by standard wet etching procedure leave behind mirror-like etched surface. On the basis of these experiments, a two-step process of electrochemical etching of SiC down to the depth of the thickness of the porous layer was suggested. Later on, in 2006, a similar technique of two-step deep etching of 6H-SiC was optimized for formation of membranes for prospective SiC pressure sensors [27].

The first publications by Shor et al. were generating a great deal of interest manifested in an increasing number of articles devoted to this topic. Konstantinov et al. reported their experience of anodizing *n*-type 6H-SiC in a 2.5 M aqueous NH_4F solution with and without illumination (photoassisted (PA) and dark current mode (DCM), respectively) [28,29]. The authors showed that the structural and electrical properties of the resulting material were determined by the conditions of the anodization. The electrical characteristics of the samples anodized in the dark mode were similar to those of the original 6H-SiC. SiC treated under UV illumination exhibited semi-insulating properties. A correlation was established between the lateral dimensions of the crystalline interpore space (SiC «columns» or «fibers») and resistivity of the PSC structures, and a model of self-regulation of the lateral dimensions of crystalline framework within the structures was proposed. The model relates the blocking of the process of increasing of pores diameter along with decreasing width of the columns with the effect of pinning of Fermi level at surface states at the columns walls. Furthermore, it was demonstrated that for mesa-structures of diodes with *p-n* junction with walls passivated by a semi-insulating layer, the breakdown voltage increased from 250-300 V, typical of crystalline SiC, up to 600 V [30]. This value represented about 80% of the analytically calculated maximum value of the breakdown voltage for monocrystalline 6H-SiC. Around the same time, Collins et al. also showed that the PSC could be more electrically resistive compared to the origi-

nal 6H-SiC [31]. It is important to note that in traditional models of electrochemical etching and pore formation in semiconductor materials, it is generally accepted that electron holes are necessary for the interaction of the electrolyte with the exposed material. Thus, when anodizing materials with an electronic type of conductivity, where holes are minority charge carriers, to create additional holes, it is necessary to use anodizing modes with external illumination of the sample/electrolyte interface. In contrast, the results of Konstantinov et al. showed that a porous structure in an *n*-type semiconductor material can be formed without additional illumination. A little bit later, Lagemaat et al. also reported on preparation of similar columnar structures with column sizes of the order of 50 nm based on *n*-type 6H-SiC single crystals anodized under DCM conditions [32]. Jassensky et al. demonstrated that morphology of porous samples with fiber-like structure depended strongly both on the illumination level and HF concentration [33]. W. Shin with co-workers reported that not only parameters of anodization defined the type of resulted porous structures, but also the doping level of the initial SiC crystals did [34]. Similar results of detailed micro-structural studies of PSC prepared on the basis of single-crystal SiC samples with 4H- and 6H-polytypes were published in a series of works by Zangoie et al. [35-39] and groups of researchers from the Ioffe Institute (Russia) [40-43]. On the basis of them, one generalizing conclusion can be drawn. Namely, porous structures prepared in aqueous solutions of hydrofluoric acid with ethyl alcohol demonstrated a presence of a disordered phase on the pores walls. Chemical composition of such a phase has deviation from stoichiometric composition of the SiC single crystal. Such a phase, presumably amorphous, is enriched with carbon and/or silicon. The exact cause of these local changes in the PSC structures has not been established.

Yet another batch of reports on preparation of porous SiC with both *n*- and *p*-type conductivity by using electrolyte with an addition of ethanol was presented by Y. Shihskin et al. from the University of Pittsburgh. They are the only group that reported on huge variation in porous structure morphology formed under various anodization conditions on the basis of 4H- and 6H-SiC with various orientations, and on the basal plane of 3C-SiC. Some properties of the structures were also discussed [44-48], and the empirical models for the formation of particular types of porous structures were proposed. According to the authors, under UV illumination crystallographic planes terminated with silicon atoms are more resistant to electrolytic attack than the planes terminated with carbon and mixed silicon-carbon atoms. This results in the formation of so-called «triangular-channel pore morphologies» in 4H-SiC, which they

observed. Later, they reported on studying the formation of a columnar mesoporous structure in single-crystalline *n*-type 6H-SiC. The different experimental conditions for both Si- and C-face of SiC samples were discussed [49].

From the other hand, it was shown that anodic treatment of *n*-type SiC can result in formation of highly porous yet amorphous anodic films along with accumulation of the products of electrochemical reaction within the porous structures [50,51]. Detailed study of the properties of such films was done in [51]. According to the Auger electron spectroscopy data, 5±0.5 at.% average deviation from the starting material stoichiometry towards elemental carbon enrichment was determined within the anodic films formed in HF-based electrolytes with ethanol in several concentrations. In addition, an elemental analysis performed with electron diffraction spectroscopy showed that the films were enriched with oxygen and fluorine at the level of (5–7) at.% and (7–11) at.%, respectively. It was found that the samples with an increased content of detectable chemical impurities had pronounced photosensitivity. The degree of observed photosensitivity in these samples is determined by the concentration of the products of chemical reactions on the surface or in the bulk of the material. It is well-known that intermediates can be responsible for photocurrent multiplication at illuminated semiconductor/fluorine-containing electrolyte junction [52,53]. For example, there is a phenomenon of photocurrent doubling during electrochemical treatment of Si with fluorine-containing solutions, which is associated with the formation of intermediate SiF_x complexes, originating from multiple charge-transfer reaction capable to inject electrons into semiconductor.

At this point of the review, a couple of comments should be made that may help the reader to adequately assess already published results or these of his/her own. Considered together, the above results show that not all the porous phases that form as a result of anodization of SiC really have something to do with silicon carbide. Highly likely, these could be porous structures based on SiC with pronounced photosensitive properties reported in [54,55], and structures with an unusual variety of morphologies. The latter statement is confirmed by the results of experiments on anodization of amorphous SiC layers deposited by plasma-enhanced chemical vapor deposition on a single-crystalline Si substrate presented in [56], where a wide variety of morphologies of porous structures in amorphous SiC were observed. Yet another example can be given, as the conclusions made in Ref. [57] can also be referred to as ambiguous. In that work, the localization of pores formed in the bulk of SiC was linked to the location of structural defects in the initial material. However, the spatial scales of distri-

bution of the pores and extended defects in the commercial SiC wafers used in that work (at the time of research, at a level of 10^2 – 10^3 cm⁻² [58,59]) look incommensurable. It should be noted also that in the overwhelming majority of publications concerning the preparation and study of the properties of porous SiC, the area of the initial SiC samples is 1–4 cm². Typically, these samples are prepared by division into parts of industrial platelets. For their anodization, homemade electrochemical cells of various designs were used. In this regard, it is obvious that the results presented by different research groups have a certain degree of uniqueness. This not only makes them difficult to reproduce, but also often makes it difficult to generalize.

The first report on advances in the development of porous SiC over the entire area of commercial 2-inch wafers was made in 2000 at the European conference “Silicon Carbide and Related Materials” (ECSCRM2000) held at Kloster Banz, (Germany). The reported results excited interest of scientific community and were highlighted in a follow-up conference digest published in Compound Semiconductors periodic magazine [60].

The most complete and consistent studies were performed within the effort undertaken to develop porous structures based on wide-gap materials SiC and GaN (the latter was obtained for the first time) within the framework of the program “Nanoporous templates for reducing large defects in SiC and GaN, nanocatalysis, magnetic clusters and biotechnology”. As part of these studies with broad international participation, a comprehensive characterization of the properties of porous SiC structures was carried out using almost all available methods for assessing their chemical composition and quality of the crystal structure, electrical and optical properties, as well as the stability of the studied properties under external influence [61–72].

Large area porous structures were obtained from the then commercially available 4H- and 6H-SiC wafers with a diameter of 2 inches with *n*-type conductivity. This material was conceived as the main substrate material (porous substrates) for homo- and heteroepitaxial growth studies using a wide range of growth technologies. As part of this work, a technology was developed that allowed for producing porous layers only on a portion of the wafers. Taking into account the existing heterogeneity of the properties of the initial material over the area, it was necessary to have such samples to compare the properties of the obtained porous structures with the properties of the initial SiC from the same plate [69].

It was found that porous structures retaining the stoichiometry of SiC were formed in the range of anodizing current densities of 4–80 mA/cm². It was shown that the nanoporous structure of SiC was a “primary”

porous structure. It was also shown that microporous structure by its genesis was a "secondary" one, and resulted from the coarsening of the nanoporous structure by the classical mechanism of Ostwald ripening [73,74]. The experimental results on two types of porous structure formation in SiC were later duplicated in Ref. [75]. The coarsening of porous structures is a self-organized process of coarsening of pores without changing their percentage of retention in the monocrystalline volume of the material. The driving force of the structural coarsening is the tendency of multiphase systems to decrease the internal free energy associated with the interphase boundaries (in the case under consideration, the surfaces of the pore walls). A vacancy-diffusion mechanism was proposed that described the contribution of physical self-organization processes to the formation of secondary porous structures with different morphology during SiC anodization [71]. A characteristic feature of the obtained porous structures of both types was the presence of a single-crystal subsurface layer with a thickness of about 30 nm, which did not contain pore channels. It has been suggested that the formation of such a dense layer is manifestation of the coarsening of the porous structure as well.

When using anodizing currents with a density more than 80 mA/cm², it was observed that degradation of the surface of the treated samples resulted in formation of fiber structures. The data of studies of the elemental composition of these SiC samples showed the presence of surface phases with a high carbon content (C = 74%, Si = 26%). In addition, high-resolution transmission electron microscopy studies revealed the presence of a disordered phase with high carbon content on the walls of fiber-like crystallites (for more details, see [68]).

3. THERMAL STABILITY OF POROUS SiC STRUCTURES

A number of works discussed the possibility of applying to porous SiC structures the conditions of technological processes that are usually used in the technology of semiconductor materials. It was established that a nanoporous structure retains the chemical composition of SiC during annealing in an Ar atmosphere up to a temperature of 1700°C, and annealing in a vacuum, up to a temperature of 1500 °C. In microporous structures, the stoichiometry of SiC is retained up to a heating temperature of 1500 °C in an argon atmosphere, under vacuum conditions, up to a temperature of 1200 °C [72]. Ignorance of these features of the behavior of porous SiC structures under conditions of high-temperature pre-epitaxial heating can lead to undesirable graphitization of the substrate surface, as discussed in [77]. The results of the epitaxial growth of SiC by sublimation in

vacuum at a temperature of 2000 °C presented in that work indicated that the conditions for processing such substrates before growth, especially those that require heating, must be adjusted in order to avoid thermal degradation of the substrate surface leading to graphitization and, as a consequence, poor quality of epitaxy. This is confirmed by the results of Refs. [72,76], where it was shown that the suppression of surface decomposition of porous SiC structures upon heating to much higher temperatures (more than 2200 °C) is possible when carrying out annealing under certain conditions, which are provided, for example, by a closed isothermal cell. Also, it is important to correctly choose the type of porous structure in terms of the stability of its properties depending on the temperature and ambient of the treatments. It was demonstrated that growth methods using substrates with a nanostructured volume and with pore-free subsurface layer had an obvious advantage for improving the quality of both homo- and heteroepitaxy of SiC and GaN.

4. ACHIEVEMENTS IN EPITAXIAL GROWTH OVER POROUS SUBSTRATES

During the period of the development of porous semiconductor technology considered in this review, most attention was paid to the possibility of controlling the quality of epitaxial growth using substrate engineering methods. One of the concepts of these methods was based on achievement of control over mismatch stresses value in heteroepitaxial films using compliant substrates. Another approach, nano-heteroepitaxy, was considered as promising for reducing both the mismatch stresses and for preventing the propagation of defects from substrates into epitaxial layers [78-82]. Both approaches to controlling the level of defects and structural stresses have also been used for epitaxial growth on porous SiC substrates; see, for example, papers on GaN-on-SiC heteroepitaxy. To use the concept of nanoheteroepitaxy, various methods of pre-treatment of porous substrates were used to remove a dense layer and thus create a growth surface with open porosity [83-88].

The best results in improving the epitaxial growth of SiC and GaN have been demonstrated on an already anodized substrate surface without any additional processing. The first reports were dated by 1999-2000. The presence of a dense layer on the surface of such a substrate material has been found to be a clear advantage [89-92].

It has also been proven that a substrate with porous layers can indeed serve as a compliant one. It should be noted that there were no GaN substrates available at that time. In heteroepitaxial layers lattice mismatch

inevitably leads to the appearance of dislocations. In GaN layers grown by chloride hydride epitaxy (HVPE) on SiC substrates with porous structures the density of threading dislocations was two orders of magnitude lower than that in layers grown on a conventional wafer. Experimentally observed manifestations of the effect of the portioning of the misfit stresses between porous substrates and the layers vs. dislocation density is discussed in Refs [91, 92].

The first achievements in improving the homoepitaxial growth of SiC and GaN by chemical vapor deposition (CVD) [93-95] and HVPE on substrates with a nanostructured volume were reported in 2000 and 2013, accordingly. When studying the properties of homoepitaxial layers, the mechanism of collective interaction in a system of point and linear structural defects in the porous structure of substrates was revealed for the first time. This unique self-regulating phenomenon determined the achievement of the goal of eliminating defects in the layers, including those emanating from the substrates [60,96,97]. The improvement in the quality of the structure of epitaxial layers on porous substrates was confirmed by the results of studying the superb characteristics of Schottky diodes based on such SiC homoepitaxial structures [98, 99]. A theoretical analysis of the behavior of extended defects (micropipes and threading dislocations) in porous media of SiC and GaN was carried out in Refs. [100-104]. These studies supported the statement that pores within the substrate may help reducing dislocations or strain, thereby improving the properties of epitaxial layers.

5. DOPANT DIFFUSION IN POROUS SiC

Another striking example of expanding the possibilities offered by the use of porous structures in the field of traditional technologies of microelectronics is the demonstration of the possibility of their advantages in diffusion doping. As is well known, wide-gap semiconductors GaN and SiC are difficult to dope with diffusion. The diffusion coefficients and solubility limits of dopants in SiC are very low even at temperatures above 1800 °C. For these reasons, traditional diffusion technologies are ineffective. In a series of works, it was shown that porous structures were an effective medium for diffusion doping of both materials, and that the effective diffusion coefficients in porous SiC and GaN are much higher than the tabulated diffusion coefficients in non-porous forms of these materials [105-109].

It was found that in porous SiC the mechanisms of impurity diffusion at low (1100–1400 °C) and high (1700–2200 °C) temperatures are fundamentally different. At low temperatures, diffusion has a surface character due

to the existence of accelerated diffusion paths, which are the internal free surfaces of the pore channels. The results of modeling high-temperature diffusion of impurities in porous SiC showed that if diffusion processes and coarsening of the porous structure via spheroidization of the pore channels occur simultaneously, diffusion with concentration-dependent coefficient is provided by the appearance of flows of internal non-equilibrium vacancies [108]. High-temperature diffusion in porous SiC was also studied in [109]. It was concluded that the diffusing impurity are transferred into the bulk through the gas phase formed within the pore channels. However, this proposition did not allow for providing an adequate explanation for the obtained values of the solubility of dopants achieved in that work.

The knowledge gained about the mechanisms of diffusion made it possible to propose practically important methods for doping wide-gap semiconductors. It is shown that low-temperature diffusion of a compensating impurity, such as vanadium, in SiC makes it possible to fabricate semi-insulating layers demanded for power and high-frequency electronic devices based on SiC, and that a similar effect could be achieved by introducing deep levels provided by silicon vacancies [105,106]. Efficient doping of GaN with an acceptor impurity directly during epitaxial growth has been demonstrated using porous SiC substrates implanted with magnesium [107]. It was also shown that the dopant can be introduced from a chemical solution deposited on the surface of porous SiC structures. This method applied to carry out low-temperature diffusion of erbium impurity has prospects for infrared light-emitting structures [110].

The developing platform of technologies for creating porous structures opens up new opportunities for the development of interdisciplinary areas of the use of semiconductor materials. For example, it has been shown that new functional materials with hierarchical structures consisting of elements of different scales can be obtained on the basis of SiC structures, which are relevant, in particular, for bioengineering applications [111]. Note that the prospects for the use of porous silicon carbide in the field of bioengineering have been studied since the first decade of the 2000s. The main achievements in this field are summarized in the one of the first reviews devoted to advances in this area, which discusses the biofunctionality of SiC and porous structures based on it for the development of biosensors, implants of various living tissues, membranes for microdialysis as well as biomedical devices [112].

6. CONCLUSION

Thus, it is obvious, that the considered early works have accumulated basic knowledge in the field of the

development of new functional materials based of porous semiconductors. In particular, it was shown that one of the prospective ways to reach a new level of functionality of semiconducting SiC was possible via formation of porous structures. It was shown that the effectiveness of such an approach in a number of cases significantly exceeds the effectiveness of traditional technologies, which follows from the collection of the reviewed results.

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