

# MATERIAŁY

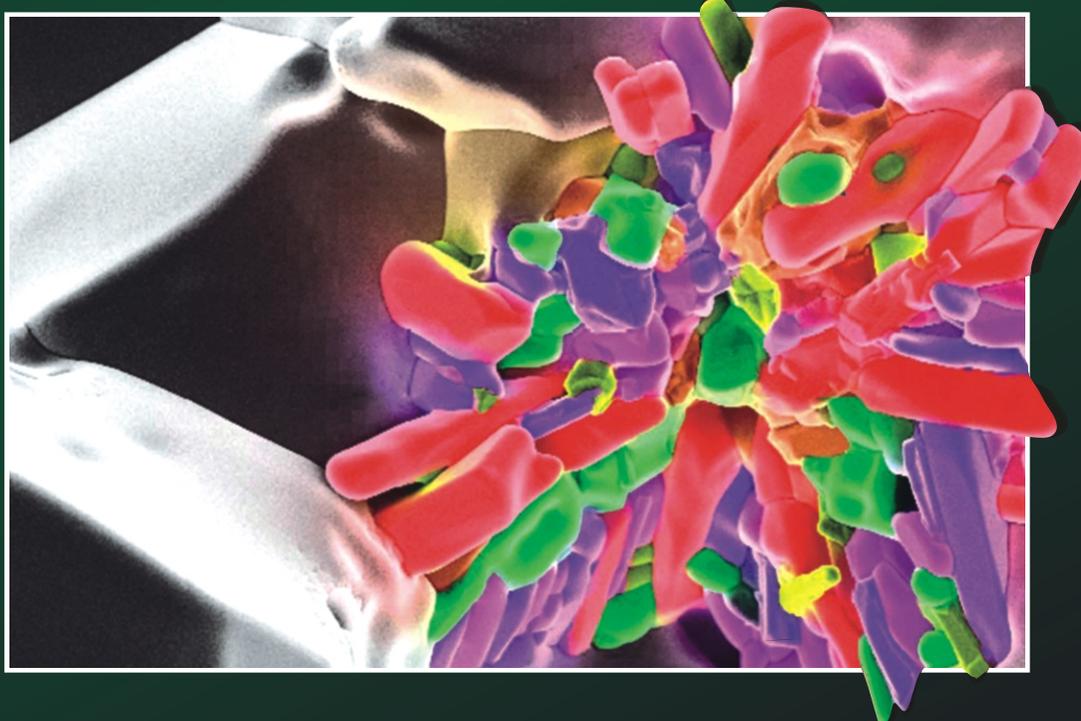
PL ISSN 0209-0058

# ELEKTRONICZNE

## ELECTRONIC MATERIALS

# 2

Tom 44  
Rok 2016



K. Pietrzak, A. Gładki,  
K. Frydman, D. Wójcik-Grzybek,  
K. Kaszyca, P. Borkowski

**Electrical properties of Ag-C and Cu-C  
contact materials**

4

I. Józwik

**Scanning electron microscope at low voltage  
operation – a unique characterization tool  
for graphene layers**

11

K. Kielbasiński, J. Szalapak,  
J. Krzemiński, A. Młodziak,  
M. Jakubowska, S. Szostak

**Stabilność wysokotemperaturowa ekologicznych  
rezystorów grubowarstwowych**

17



INSTYTUT TECHNOLOGII MATERIAŁÓW ELEKTRONICZNYCH  
INSTITUTE OF ELECTRONIC MATERIALS TECHNOLOGY

<http://rcin.org.pl>



**INSTYTUT TECHNOLOGII  
MATERIAŁÓW ELEKTRONICZNYCH**  
ul. Wólczyńska 133, 01-919 Warszawa

**Redaktor Naczelny**  
tel.: (+48 22) 834 90 03  
(+48 22) 639 58 05  
e-mail: [itme@itme.edu.pl](mailto:itme@itme.edu.pl)

**Dział Informacji Naukowej  
i Technicznej**  
tel.: (+48 22) 639 55 29  
e-mail: [ointe@itme.edu.pl](mailto:ointe@itme.edu.pl)  
[www.itme.edu.pl](http://www.itme.edu.pl)

Instytut Technologii Materiałów Elektronicznych wydaje dwa czasopisma naukowe, których tematyka dotyczy inżynierii materiałowej, elektroniki i fizyki ciała stałego, a w szczególności technologii otrzymywania nowoczesnych materiałów, ich obróbki, miernictwa oraz wykorzystania dla potrzeb elektroniki i innych dziedzin gospodarki:

- **Materiały Elektroniczne** – zawierające artykuły problemowe, teksty wystąpień pracowników ITME na konferencjach i Biuletyn PTWK,
  - **Prace ITME** – zawierające monografie, rozprawy doktorskie i habilitacyjne
- oraz
- stale aktualizowane katalogi i karty katalogowe technologii, materiałów, wyrobów i usług oferowanych przez Instytut i opartych o wyniki prowadzonych prac badawczych, opisy nowych wyrobów, metod i aparatury

Informacje można uzyskać:

**Dział Organizacji** tel.: (48 22) 639 58 08  
**Promocja i Marketing** tel.: (48 22) 639 58 32  
e-mail: [itme@itme.edu.pl](mailto:itme@itme.edu.pl)

INSTYTUT TECHNOLOGII MATERIAŁÓW ELEKTRONICZNYCH

**MATERIAŁY  
ELEKTRONICZNE  
ELECTRONIC MATERIALS  
KWARTALNIK**

**T. 44 - 2016 nr 2**

Wydanie publikacji dofinansowane jest przez  
Ministerstwo Nauki i Szkolnictwa Wyższego

WARSZAWA ITME 2016

<http://rcin.org.pl>

## KOLEGIUM REDAKCYJNE

### Redaktor Naczelny:

dr inż. Ireneusz MARCINIAK

### Redaktorzy tematyczni:

#### Z-ca Red. Naczelnego

dr hab. inż. Katarzyna PIETRZAK, prof. ITME

prof. dr hab. inż. Andrzej JELEŃSKI

dr hab. inż. Paweł KAMIŃSKI, prof. ITME

dr hab. Dorota PAWLAK, prof. ITME

dr inż. Włodzimierz STRUPIŃSKI

prof. dr hab. inż. Andrzej TUROS

### Rada programowa:

prof. dr hab. Jacek BARANOWSKI

prof. dr hab. inż. Zbigniew BIELECKI

prof. dr hab. Marek GODLEWSKI

prof. dr hab. Maria KAMIŃSKA

dr hab. inż. Jarosław MIZERA, prof. PW

prof. dr hab. inż. Antoni ROGALSKI

### Sekretarz redakcji:

mgr Anna WAGA

### Redaktorzy językowi:

mgr Maria SIWIK - GRUŻEWSKA

mgr Krystyna SOSNOWSKA

### Redaktor techniczny:

mgr Szymon PLASOTA

## ADRES REDAKCJI

**Instytut Technologii Materiałów Elektronicznych**

ul. Wólczyńska 133, 01-919 Warszawa,

e-mail: ointe@itme.edu.pl;

www: matelektron.itme.edu.pl

## KONTAKT

### redaktor naczelny:

tel.: (22) 834 90 03 oraz (22) 639 58 05

sekretarz redakcji: (22) 639 55 29

PL ISSN 0209 - 0058

**Kwartalnik notowany na liście czasopism naukowych**

**Ministerstwa Nauki i Szkolnictwa Wyższego**

7 pkt. - wg komunikatu MNiSW.

**Opublikowane artykuły są indeksowane w bazach**

**danych:** BazTech, CAS - Chemical Abstracts oraz In-

dex Copernicus

**Publikowane artykuły mające charakter naukowy są**

**recenzowane przez samodzielnych pracowników na-**

**ukowych.**

**Wersja papierowa jest wersją pierwotną.**

**Kwartalnik publikowany jest w otwartym dostępie.**

**Nakład: 200 egz.**

**Na okładce:** ceramika  $Y_2O_3$

**Autor zdjęcia:** dr inż. Anna Piątkowska

**Projekt:** „Właściwości mechaniczne i odporność na szoki termiczne przezroczystej ceramiki  $Y_2O_3$  w funkcji mikrostruktury i temperatury”, finansowany przez Narodowe Centrum Nauki.

**Koordynator projektu:** dr Marek Boniecki

## SPIS TREŚCI - CONTENTS

Electrical properties of Ag-C and Cu-C contact materials	K. Pietrzak, A. Gładki, K. Frydman, D. Wójcik-Grzybek, K. Kaszyca, P. Borkowski	4
Właściwości elektryczne materiałów stykowych Ag-C i Cu-C		
Scanning electron microscope at low voltage operation – a unique characterization tool for graphene layers	I. Jóźwik	11
Skaningowy mikroskop elektronowy pracujący w zakresie niskich wartości napięcia przyspieszającego jako unikatowe narzędzie do charakteryzacji warstw grafenu		
Stabilność wysokotemperaturowa ekologicznych rezystorów grubowarstwowych	K. Kielbasiński, J. Szałapak, J. Krzemiński, A. Młodziak, M. Jakubowska, S. Szostak	17
High temperature stability of eco-friendly thick-film resistors		
Streszczenia wybranych artykułów pracowników ITME		27

# STRESZCZENIA ARTYKUŁÓW ME 44 - 2 - 2016

## Właściwości elektryczne materiałów stykowych

### Ag-C i Cu-C

ME 44, 2, 2016, s. 4

Przemysłowe otrzymywanie rozmaitych form węgla – grafenu, nanorurek, fulerenów – rozszerzyło gamę materiałów kompozytowych, w których stanowią one fazę wzmacniającą matryc metalicznych. Oczekiwano, że grafenowa faza wzmacniająca polepszy cechy elektryczne, cieplne i wytrzymałościowe takich kompozytów. Szczególnie obiecującymi są kompozyty z matrycami Cu lub Ag znajdujące zastosowania w mikroelektronice i optoelektronice, przemyśle lotniczym i samochodowym. Specyficzna grupa tych kompozytów stosowana jest w rozmaitych elementach układów elektrycznych jako wyłączniki etc. Do grupy tej należą między innymi kompozyty Ag-W; Ag-WC, Ag-WC-C lub Cu-W. Prezentowane wyniki badań elektrycznych kompozytów Cu(Ag)/GF poszerzają zbiór właściwości materiałów stosowanych w powietrznych i próżniowych stykach elektrycznych.

## Skaningowy mikroskop elektronowy pracujący w zakresie niskich wartości napięcia przyspieszającego jako unikatowe narzędzie do charakteryzacji warstw grafenu

ME 44, 2, 2016, s. 11

Próbki grafenu otrzymywanego metodą chemicznego osadzania z fazy gazowej na podłożach Cu poddano badaniom przy użyciu komercyjnego skaningowego mikroskopu elektronowego (SEM) pracującego w zakresie niskich wartości napięcia przyspieszającego. Optymalizacja warunków obrazowania SEM prowadzona w trybie dwukanałowym (rejestracja obrazów SE1 i BSE podczas tego samego skanu) pozwoliła na wizualizację typowych cech grafenu na Cu takich jak fałdy, pęknięcia i dodatkowe warstwy. Mechanizm kontrastu związanego ze zmianami liczby warstw grafenu obserwowanego w obrazach BSE został przedstawiony w oparciu o detekcję elektronów BSE o niskich stratach energii. Przeciwwytleniające właściwości grafenu na metalicznych podłożach zostały potwierdzone poprzez możliwość obserwacji kontrastu kanałowania elektronów w podłożach Cu przy energii elektronów pierwotnych rzędu 0,5 keV.

## Stabilność wysokotemperaturowa ekologicznych rezystorów grubowarstwowych

ME 44, 2, 2016, s. 17

W związku z Dyrektywą RoHS, która weszła w życie w 2006 roku producenci materiałów elektronicznych zostali zmuszeni do wyeliminowania m.in. ołowiu i jego związków ze składów tych materiałów. Dyrektywa wymusiła zmianę składu past służących do wytwarzania rezystorów grubowarstwowych szeroko rozpowszechnionych w konsumenckim sprzęcie elektronicznym. Autorzy zaproponowali nowatorskie pasty rezystywne zdolne w pełni zastąpić pasty oparte o ołów i jego związki. Ponadto zostały przeprowadzone badania odporności na narażenia termiczno-prądowe, które ujawniły przewagę użytkową w aspektach stabilności rezystancji na cykliczne narażenia temperaturowe nowych past wobec past tradycyjnych.

# THE ARTICLES ABSTRACTS ME 44 - 2 - 2016

## Electrical properties of Ag-C and Cu-C contact materials

ME 44, 2, 2016, p. 4

Industrial production of various forms of carbon, including graphene, nanotubes, and fullerenes, expanded the range of composite materials for which they constitute the reinforcing phase of metallic matrices. It was expected that the graphene form (GF) reinforcing phase would improve the electrical, thermal, and mechanical properties of such composites. Composites with Cu and Ag matrices, having a wide range of applications in micro- and optoelectronics, aerospace and automotive industries, proved to be particularly promising. A specific group of these composites is used in a variety of electrical circuits for electrical switches, contactors, circuit breakers, voltage regulators, and arcing tips. Among others, this group includes composites such as Ag-W, Ag-WC, Ag-WC-C, or Cu-W. The presented results of electrical tests performed for the Cu (Ag) /GF composites extend the number of properties of materials used in air and vacuum electrical contacts.

## Scanning electron microscope at low voltage operation – a unique characterization tool for graphene layers

ME 44, 2, 2016, p. 11

Graphene grown on Cu foils by chemical vapor deposition (CVD) technique has been investigated using commercially available scanning electron microscope at low voltage operation. The optimized conditions of SEM imaging carried out in a double-channel mode (registering secondary electrons type 1 (SE1) and backscattered electrons (BSE) images in a single scan) allowed for the visualization of typical features of graphene on Cu, such as folds, cracks and add-layers. The mechanism of thickness contrast observed in the BSE images was described in terms of low loss-BSE detection. Antioxidant qualities of graphene sheets on metallic substrate were confirmed by the ability of observation of the channeling contrast in Cu substrates at primary electrons energy of 0.5 keV.

## High temperature stability of eco-friendly thick-film resistors

ME 44, 2, 2016, p. 17

Under the EU directive on the Restriction of Hazardous Substances (RoHS) implemented in 2006, producers of electronics materials were obliged to eliminate lead and its compounds from the composition of their products. As a consequence of this directive the changes affected the compositions of pastes used in the production of thick-film resistors, widely used in mass electronics. The authors have developed new compositions for the resistive thick film pastes, to replace the ones based on lead. Moreover, the thermal and electrical studies showed the advantage of the new compositions over the traditional ones in terms of resistance stability under temperature cyclic test.

## STRESZCZENIA WYBRANYCH ARTYKUŁÓW PRACOWNIKÓW ITME

### **Polyelectrolyte multilayer film modification for chemo-mechano-regulation of endothelial cell response**

**Mzyk A.<sup>1</sup>, Lackner J. M.<sup>2</sup>, Wilczek P.<sup>3</sup>, Lipińska L.<sup>4</sup>, Niemiec-Cyganek A.<sup>3</sup>, Samotus A.<sup>3</sup>, Morenc M.<sup>3</sup>**

<sup>1</sup> Institute of Metallurgy and Materials Science, Polish Academy of Sciences, 25 Reymonta Street, 30-059 Krakow, Poland

<sup>2</sup> Joanneum Research Forschungsges mbH, Institute of Surface Technologies and Photonics, Functional Surfaces, Leobner Strasse 94, A-8712 Niklasdorf, Austria

<sup>3</sup> Foundation for Cardiac Surgery Development, Wolności Street 345a, 41-800 Zabrze, Poland

<sup>4</sup> Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

*RSC Advances*, 2016, 6, 11, 8811 - 8828

The new multilayer polyelectrolyte films (PEMs) that are able to simulate the structure and functions of the extracellular matrix have become a powerful tool for tailoring biointerfaces of implants. In this study, bioactive PEM coatings have been investigated as a supportive system for efficient endothelialization of cardiovascular implants. The modern films were designed in a manner that allows one to potentially induce specific response from the tissues surrounding the biomaterial due to its chemical composition as well as mechanical properties. The PEM rigidity was regulated by the cross-linking chemistry as well as nanoparticle incorporation, while biochemical modification was performed by the VEGF adsorption within coatings. Obtained results have shown that PEM/VEGF films enhanced in vitro spreading and proliferation of endothelial cells, whereas VEGF presence inhibited IL-6 production and release. Since non-functionalized films also contributed to proliferation of endothelial cells and cytokine secretion, it may be supposed that PEM stiffness acts in synergy with the growth factor, but probably through a different pathway. Results clearly demonstrate the effectiveness of the proposed endothelialization strategy and confirm correlation between the chemical and mechanical properties of the PEMs in vitro.

### **‘Clickable’ ZnO nanocrystals: the superiority of a novel organometallic approach over the inorganic sol–gel procedure**

**Grala A.<sup>1,2</sup>, Wolska-Pietkiewicz M.<sup>1</sup>, Danowski W.<sup>1</sup>, Wróbel Z.<sup>2</sup>, Grzonka J.<sup>2,3,4</sup>, Lewiński J.<sup>1,2</sup>**

<sup>1</sup> Warsaw University of Technology, Faculty of Chemistry, Noakowskiego 3, 00-664 Warsaw, Poland

<sup>2</sup> Polish Academy of Sciences, Institute of Physical Chemistry,

Kasprzaka 44/52, 01-224 Warsaw, Poland

<sup>3</sup> Warsaw University of Technology, Faculty of Materials Science and Engineering, Wołoska 141, 02-507 Warsaw, Poland

<sup>4</sup> Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

*Chemical Communications*, 2016, 52, 7340 - 7343

We demonstrate for the first time a highly efficient Cu(I)-catalyzed alkyne–azide cycloaddition reaction on the surface of ZnO nanocrystals with retention of their photoluminescence properties. Our comparative studies highlight the superiority of a novel self-supporting organometallic method for the preparation of brightly luminescent and well-passivated ZnO nanocrystals over the traditional sol–gel procedure.

### **Large-area high-quality graphene on Ge(001)/Si(001) substrates**

**Pasternak I.<sup>1</sup>, Dabrowski P.<sup>2</sup>, Ciepiewski P.<sup>1</sup>, Kolkovsky V.<sup>3</sup>, Klusek Z.<sup>2</sup>, Baranowski J. M.<sup>1</sup>, Strupinski W.<sup>1</sup>**

<sup>1</sup> Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

<sup>2</sup> Department of Solid States Physics, University of Lodz, Pomorska 149/153, Lodz, Poland

<sup>3</sup> Institute of Physics, Polish Academy of Sciences, Al. Lotnikow 32/46, Warsaw, 02-668 Poland

*Nanoscale*, 2016, 8, 11241 - 11247

Various experimental data revealing large-area high-quality graphene films grown by the CVD method on Ge(001)/Si(001) substrates are presented. SEM images have shown that the structure of nano-facets is formed on the entire surface of Ge(001), which is covered by a graphene layer over the whole macroscopic sample surface of 1 cm<sup>2</sup>. The hill-and-valley structures are positioned 90° to each other and run along the <100> direction. The hill height in relation to the valley measured by STM is about 10 nm. Raman measurements have shown that a uniform graphene monolayer covers the nano-facet structures on the Ge(001) surface. Raman spectroscopy has also proved that the grown graphene monolayer is characterized by small strain variations and minimal charge fluctuations. Atomically resolved STM images on the hills of the nanostructures on the Ge(001) surface have confirmed the presence of a graphene monolayer. In addition, the STS/CITS maps show that high-quality graphene has been obtained on such terraces. The subsequent coalescence of graphene domains has led to a relatively well-oriented large-area layer. This is confirmed by LEED measurements, which have indicated that two orientations are preferable in the grown large-area graphene monolayer. The presence of large-area coverage by graphene has been also confirmed by low temperature Hall

measurements of a macroscopic sample, showing an n-type concentration of  $9.3 \times 10^{12} \text{ cm}^{-2}$  and a mobility of  $2500 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ . These important characteristic features of graphene indicate a high homogeneity of the layer grown on the large area Ge(001)/Si(001) substrates.

### Optical-quality controllable wet-chemical doping of graphene through a uniform, transparent and low-roughness F4-TCNQ/MEK layer

Misseeuw L.<sup>1</sup>, Krajewska A.<sup>2,3</sup>, Pasternak I.<sup>3</sup>, Ciuk T.<sup>3</sup>, Strupinski W.<sup>3</sup>, Reekmans G.<sup>4</sup>, Adriaensens P.<sup>4</sup>, Geldof D.<sup>5</sup>, Blockhuys F.<sup>5</sup>, Van Vlierberghe S.<sup>1,5,6</sup>, Thienpont H.<sup>1</sup>, Dubruel P.<sup>6</sup>, Vermeulen N.<sup>1</sup>

<sup>1</sup> Brussels Photonics Team (B-PHOT), Dept. of Applied Physics and Photonics (IR-TONA), Vrije Universiteit Brussel, Pleinlaan 2, B-1050 Brussels, Belgium

<sup>2</sup> Institute of Optoelectronics, Military University of Technology, Gen. S. Kaliskiego 2, 00-908 Warsaw, Poland

<sup>3</sup> Institute of Electronic Materials Technology, Wolczynska 133, 01-919 Warsaw, Poland

<sup>4</sup> Applied and Analytical Chemistry, Institute for Materials Research (IMO), Hasselt University, Agoralaan 1-Building D, B-3590 Diepenbeek, Belgium

<sup>5</sup> Department of Chemistry, University of Antwerp, Groenenborgerlaan 171, B-2020 Antwerp, Belgium

<sup>6</sup> Polymer Chemistry & Biomaterials Research Group, Ghent University, Krijgslaan 281 (S4 Bis), B-9000 Ghent, Belgium

*RSC Advances*, 2016, 6, 104491 - 104501

Controllable chemical doping of graphene has already proven very useful for electronic applications, but when turning to optical and photonic applications, the additional requirement of having both a high transparency and a low surface roughness has, to our knowledge, not yet been fulfilled by any chemical dopant system reported so far. In this work, a new method that meets for the first time this optical-quality requirement while also providing efficient, controllable doping is presented. The method relies on F4-TCNQ dissolved in methyl ethyl ketone (MEK) yielding a uniform deposition after spin coating because of an extraordinary charge transfer interaction between the F4-TCNQ and MEK molecules. The formed F4-TCNQ/MEK layer exhibits a very high surface quality and optical transparency over the visible-infrared wavelength range between 550 and 1900 nm. By varying the dopant concentration of F4-TCNQ from 2.5 to 40 mg ml<sup>-1</sup> MEK, the doping effect can be controlled between  $\Delta n = +5.73 \times 10^{12} \text{ cm}^{-2}$  and  $+1.09 \times 10^{13} \text{ cm}^{-2}$  for initially strongly p-type hydrogen-intercalated graphene grown on 6H-silicon-carbide substrates, and between  $\Delta n = +5.56 \times 10^{12} \text{ cm}^{-2}$  and  $+1.04 \times 10^{13} \text{ cm}^{-2}$  for initially weakly p-type graphene transferred on silicon samples. This is the first time that truly optical-quality chemical doping of graphene is demonstrated, and the obtained doping values exceed those reported before for F4-TCNQ-based graphene doping by as much as 50%.

### Printed HF antennas for RFID on-metal transponders

Janeczek K.<sup>1</sup>, Arazna A.<sup>1</sup>, Salski B.<sup>2</sup>, Lipiec K.<sup>1</sup>, Jakubowska M.<sup>3,4</sup>

<sup>1</sup> Tele and Radio Research Institute, Warsaw, Poland

<sup>2</sup> Institute of Radioelectronics, Warsaw University of Technology, Warsaw, Poland

<sup>3</sup> Institute of Electronic Materials Technology, Warsaw, Poland

<sup>4</sup> Institute of Metrology and Biomedical Engineering, Warsaw University of Technology, Warsaw, Poland

*Circuit World*, 2016, 42, 1, pp.2

**Purpose** - The purpose of this paper is to investigate screen-printed high-frequency (HF) antennas for radio frequency identification (RFID) on-metal transponders in which a magnetic sheet was used as a substrate material.

**Design/methodology/approach** - A transponder antenna was designed in the form of square coil using a high-frequency electromagnetic software. Then, the antenna was fabricated with screen printing technique on two different magnetic sheets (RFN4 and RFN7) and on polyethylene naphthalate (PEN) foil for comparison. Its printing was carried out with polymer pastes based on silver flakes (PM-406 and SF). Thickness, track width and spacing were examined for the antennas using digital microscope and contact profilometer. Resistance and inductance were also measured, and resonant frequency, quality factor and target values of capacitance to achieve resonant frequency of the tested antenna at 13.56 MHz were calculated. Finally, RFID chips were mounted to the prepared antennas using an isotropic conductive adhesive, and a maximum read distance was measured with a reader installed in a smartphone.

**Findings** - It was found that an antenna thickness on the magnetic sheets used was higher than on PEN foil. At the same time, surface roughness of the fabricated antennas on these sheets was revealed to be higher as well. Inductance of the measured antennas exhibited good conformity with the antenna design, but higher divergence was noticed in the measured resistance. Its lowest value was achieved when the antenna was printed with the paste PM-406 on PEN foil and the highest one when it was fabricated with the paste SF on the same substrate. This suggests that high attention needs to be paid to a polymer paste selected for antenna printing. The performed tests showed that the magnetic sheet RFN4 seems to be better substrate for on-metal transponders compared to RFN7 due to lower resistance and higher quality factor of the prepared antennas.

**Research limitations/implications** - Further investigations are required to examine mechanical and thermal durability of the HF antennas printed on the magnetic sheets.

**Practical implications** - The investigated HF antennas fabricated on magnetic sheets can find application in near field communication (NFC) transponders designed to be placed on metallic surfaces, e.g. on frames of advertising screens.

**Originality/value** - Influence of used magnetic sheets and

polymer pastes on geometry and electrical properties of HF antennas for RFID on-metal transponders was investigated. The presented investigations can be interesting for NFC/RFID designers who are involved in designing systems suitable for metallic surfaces

## Ambient Temperature Growth of Mono- and Polycrystalline NbN Nanofilms and Their Surface and Composition Analysis

Krause S.<sup>1</sup>, Afanas'ev V.<sup>2</sup>, Desmaris V.<sup>1</sup>, Meledin D.<sup>1</sup>, Pavolotsky A.<sup>1</sup>, Belitsky V.<sup>1</sup>, Lubenschenko A.<sup>2</sup>, Batrakov A.<sup>2</sup>, Rudzinski M.<sup>3</sup>, Pippel E.<sup>4</sup>

<sup>1</sup> Group for Advanced Receiver Development, Chalmers University of Technology, Gothenburg, Sweden

<sup>2</sup> Department of General Physics and Nuclear Fusion, National Research University "Moscow Power Engineering Institute", Moscow, Russia

<sup>3</sup> Institute of Electronic Materials Technology (ITME), Warsaw, Poland

<sup>4</sup> Max Planck Institute of Microstructure Physics, Halle, Germany

This paper presents the studies of high-quality 5-nm-thin NbN films deposited by means of reactive dc magnetron sputtering at room temperature. The deposition without substrate heating offers major advantages from a processing point of view and motivates the extensive composition and surface characterization and comparison of the present films with high-quality films grown at elevated temperatures. Monocrystalline NbN films have been epitaxially grown onto hexagonal GaN buffer layers (0002) and show a distinct low defect interface as confirmed by high-resolution TEM. The critical temperature  $T_c$  of the films on the GaN buffer layer reached 10.4 K. Furthermore, a polycrystalline structure was observed on films grown onto Si (100) substrates, exhibiting a  $T_c$  of 8.1 K, albeit a narrow transition from the normal to the superconducting state. X-ray photoelectron spectroscopy and reflected electron energy loss spectroscopy verified that the composition of NbN was identical irrespective of applied substrate heating. Moreover, the native oxide layer at the surface of NbN has been identified as NbO<sub>2</sub> and, thus, is in contrast to the Nb<sub>2</sub>O<sub>5</sub>, usually claimed to be formed at the surface of Nb when exposed to air. These findings are of significance since it was proven the possibility of growing epitaxial NbN onto GaN buffer layer in the absence of high temperatures, hence paving the way to employ NbN in more advanced fabrication processes involving a higher degree of complexity. The eased integration and employment of liftoff techniques could, in particular, lead to improved performance of cryogenic ultrasensitive terahertz electronics.

## g-factors of conduction electrons and holes in Bi<sub>2</sub>Se<sub>3</sub> three-dimensional topological insulator

Wolos A.<sup>1,2</sup>, Szyszko S.<sup>2</sup>, Drabinska A.<sup>2</sup>, Kaminska M.<sup>2</sup>,

Strzelecka S. G.<sup>3</sup>, Hruban A.<sup>3</sup>, Materna A.<sup>3</sup>, Piersa M.<sup>3</sup>, Borysiuk J.<sup>1,2</sup>, Sobczak K.<sup>1</sup>, Konczykowski M.<sup>4</sup>

<sup>1</sup> Institute of Physics, Polish Academy of Sciences, Al. Lotnikow 32/46, 02-668 Warsaw, Poland

<sup>2</sup> Faculty of Physics, University of Warsaw, ul. Pasteura 5, 00-681 Warsaw, Poland

<sup>3</sup> Institute of Electronic Materials Technology, ul. Wolczynska 133, 01-919 Warsaw, Poland

<sup>4</sup> Laboratoire des Solides Irradiés, CNRS-UMR 7642 and CEA-DSM-IRAMIS, Ecole Polytechnique, F-91128 Palaiseau Cedex, France

*Physical Review B*, 2016, 93, 155114

Bulk-related conduction electron spin resonance and conduction hole spin resonance were investigated in Bi<sub>2</sub>Se<sub>3</sub>, a three-dimensional topological insulator. Electrons in the conduction band and holes in the valence band both have spin ½. The effective g-factors for conduction electrons are equal to  $27.3 \pm 0.15$  for magnetic field parallel to the c axis and  $19.48 \pm 0.07$  for magnetic field perpendicular to the c axis, whereas for conduction holes  $29.90 \pm 0.09$  for magnetic field parallel and  $18.96 \pm 0.04$  for magnetic field perpendicular to the c axis, respectively. Nonparabolicity effects were not observed in the investigated low carrier concentration range, below  $8 \times 10^{17} \text{cm}^{-3}$ . Large g-factors, higher by an order of magnitude than the free electron value, are due to strong spin-orbit interactions in Bi<sub>2</sub>Se<sub>3</sub>. The striking similarity of the spin resonances due to conduction electrons and holes confirms the peculiar symmetry between the conduction and valence bands of Bi<sub>2</sub>Se<sub>3</sub>, both having similar effective masses and spin character.

## Manipulating the Topological Interface by Molecular Adsorbates: Adsorption of Co-Phthalocyanine on Bi<sub>2</sub>Se<sub>3</sub>

Caputo M.<sup>1,2</sup>, Panighel M.<sup>3,2</sup>, Lisi S.<sup>4,5</sup>, Khalil L.<sup>1,6</sup>, Di Santo G.<sup>2</sup>, Papolazarou E.<sup>1</sup>, Hruban A.<sup>7</sup>, Konczykowski M.<sup>8</sup> et al.

<sup>1</sup> Laboratoire de Physique des Solides, CNRS, Université Paris-Sud, Université Paris-Saclay, 91405 Orsay Cedex, France

<sup>2</sup> Laboratory Micro & Nano-Carbon, Elettra - Sincrotrone Trieste S.C.p.A., s.s.14 Km 163.5, 34149 Trieste, Italy

<sup>3</sup> Catalan Institute of Nanoscience and Nanotechnology (ICN2), CSIC and The Barcelona Institute of Science and Technology, Campus UAB, Bellaterra, 08193 Barcelona, Spain

<sup>4</sup> Dipartimento di Fisica, Università di Roma La Sapienza, Piazzale A. Moro 5, 00185 Roma, Italy

<sup>5</sup> Institut Néel, CNRS/UGA UPR2940, 25 Rue des Martyrs BP 166, 38042 Grenoble, France

<sup>6</sup> Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP48, 91192 Gif-sur-Yvette Cedex, France

<sup>7</sup> Institute of Electronic Materials Technology, 01-919 Warsaw, Poland

<sup>8</sup> Laboratoire des Solides Irradiés, Ecole Polytechnique, CNRS, CEA, Université Paris-Saclay, 91128 Palaiseau Cedex, France

*Nano Letters*, 2016, 16, 6, 3409 - 3414

Topological insulators are a promising class of materials for applications in the field of spintronics. New perspectives in this field can arise from interfacing metal-organic molecules with the topological insulator spin-momentum locked surface states, which can be perturbed enhancing or suppressing spintronics-relevant properties such as spin coherence. Here we show results from an angle-resolved photoemission spectroscopy (ARPES) and scanning tunnelling microscopy (STM) study of the prototypical cobalt phthalocyanine (CoPc)/Bi<sub>2</sub>Se<sub>3</sub> interface. We demonstrate that the hybrid interface can act on the topological protection of the surface and bury the Dirac cone below the first quintuple layer.

### Comparative study of radiation-induced damage in magnesium aluminate spinel by means of IL, CL and RBS/C techniques

Jozwik I.<sup>1</sup>, Jagielski J.<sup>1,2</sup>, Gawlik G.<sup>1</sup>, Jozwik P.<sup>1</sup>, Ratajczak R.<sup>2</sup>, Panczer G.<sup>3</sup>, Moncoffre N.<sup>4</sup>, Wajler A.<sup>1</sup>, Sidorowicz A.<sup>1</sup>, Thome L.<sup>5</sup>

<sup>1</sup> Institute of Electronic Materials Technology Warsaw Poland

<sup>2</sup> National Centre for Nuclear Research Otwock, Swierk Poland

<sup>3</sup> Institut Lumière Matière, UMR5306 Université Lyon 1-CNRS Université de Lyon Villeurbanne Cedex France

<sup>4</sup> Institute de Physique Nucléaire Lyon, UMR5822 Villeurbanne Cedex France

<sup>5</sup> Centre de Spectrométrie Nucléaire et de Spectrométrie de Masse, IN2P3-CNRS Université Paris-Sud Orsay Cedex France

*Physics and Chemistry of Minerals*, 2016, 43, 6, 439 - 445

A comparative study of damage accumulation in magnesium aluminate spinel (MgAl<sub>2</sub>O<sub>4</sub>) has been conducted using ionoluminescence (IL), cathodoluminescence (CL) and Rutherford Backscattering Spectrometry/channeling (RBS/C) techniques. MgAl<sub>2</sub>O<sub>4</sub> single crystal and polycrystalline samples were irradiated with 320 keV Ar<sup>+</sup> ions at fluencies ranging from 1 × 10<sup>12</sup> to 2 × 10<sup>16</sup> cm<sup>-2</sup> in order to create various levels of radiation damage. RBS/C measurements provided quantitative data about damage concentration in the samples. These values were then compared to the luminescence measurements. The results obtained by IL and RBS/C methods demonstrate a two-step character of damage buildup process. The CL data analysis points to the three-step damage accumulation mechanism involving the first defect transformation at fluencies of about 10<sup>13</sup> cm<sup>-2</sup> and second at about 10<sup>15</sup> cm<sup>-2</sup>. The rate of changes resulting from the formation of nonluminescent recombination centers is clearly nonlinear and cannot be described in terms of continuous accumulation of point defects. Both, IL and CL techniques, appear as new, complementary tools bringing new possibilities in the damage accumulation studies in single- and polycrystalline materials.

### Surface and interface structure of quasi-free standing graphene on SiC

Melios C.<sup>1,2</sup>, Spencer S.<sup>1</sup>, Shard A.<sup>1</sup>, Strupiński W.<sup>3</sup>, Silva S. R. P.<sup>2</sup>, Kazakova O.<sup>1</sup>

<sup>1</sup> National Physical Laboratory, Teddington, TW11 0LW, UK

<sup>2</sup> Advanced Technology Institute, University of Surrey, Guildford, Surrey, GU2 7XH, UK

<sup>3</sup> Institute of Electronic Materials Technology, Wólczyńska 133, 01-919 Warsaw, Poland

*2D Materials*, 2016, 3, 2, 025023

We perform local nanoscale studies of the surface and interface structure of hydrogen intercalated graphene on 4H-SiC(1000). In particular, we show that intercalation of the interfacial layer results in the formation of quasi-free standing one layer graphene (QFS 1LG) with change in the carrier type from n- to p-type, accompanied by a more than four times increase in carrier mobility. We demonstrate that surface enhanced Raman scattering (SERS) reveals the enhanced Raman signal of Si-H stretching mode, which is the direct proof of successful intercalation. Furthermore, the appearance of D, D + D' as well as C-H peaks for the quasi-free standing two layer graphene (QFS 2LG) suggests that hydrogen also penetrates in between the graphene layers to locally form C-H sp<sup>3</sup> defects that decrease the mobility. Thus, SERS provides a quick and reliable technique to investigate the interface structure of graphene which is in general not accessible by other conventional methods. Our findings are further confirmed by Kelvin probe force microscopy and x-ray photoelectron spectroscopy.

### Low-noise epitaxial graphene on SiC Hall effect element for commercial applications

Ciuk T.<sup>1,2</sup>, Petruk O.<sup>3</sup>, Kowalik A.<sup>1</sup>, Jozwik I.<sup>1</sup>, Rychter A.<sup>4</sup>, Szmids J.<sup>2</sup>, Strupinski W.<sup>1</sup>

<sup>1</sup> Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

<sup>2</sup> Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland

<sup>3</sup> Industrial Research Institute for Automation and Measurements PIAP, Al. Jerozolimskie 202, 02-486 Warsaw, Poland

<sup>4</sup> Institute of Radioelectronics and Multimedia Technology, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland

*Applied Physics Letters*, 2016, 108, 223504

In this report, we demonstrate a complete Hall effect element that is based on quasi-free-standing monolayer graphene synthesized on a semi-insulating on-axis Si-terminated 6H-SiC substrate in an epitaxial Chemical Vapor Deposition process. The device offers the current-mode sensitivity of 87 V/AT and low excess noise (Hooge's parameter  $\alpha_H < 2 \times 10^{-3}$ ) enabling

room-temperature magnetic resolution of 650 nT/Hz<sup>0.5</sup> at 10 Hz, 95 nT/Hz<sup>0.5</sup> at 1 kHz, and 14 nT/Hz<sup>0.5</sup> at 100 kHz at the total active area of 0.1275 mm<sup>2</sup>. The element is passivated with a silicone encapsulant to ensure its electrical stability and environmental resistance. Its processing cycle is suitable for large-scale commercial production and it is available in large quantities through a single growth run on an up to 4-in SiC wafer.

### Strong interband Faraday rotation in 3D topological insulator Bi<sub>2</sub>Se<sub>3</sub>

Ohnoutek L.<sup>1</sup>, Hakl M.<sup>2</sup>, Veis M.<sup>1</sup>, Piot B. A.<sup>2</sup>, Faugeras C.<sup>2</sup>, Martinez G.<sup>2</sup>, Yakushev M. V.<sup>3,4</sup>, Martin R. W.<sup>3</sup>, Drasar C.<sup>5</sup>, Materna A.<sup>6</sup>, Strzelecka G.<sup>6</sup>, Hruban A.<sup>6</sup>, Potemski M.<sup>2</sup>, Orlita M.<sup>1,2</sup>

<sup>1</sup> Institute of Physics, Charles University, Ke Karlovu 5, CZ-121 16 Praha 2, Czech Republic

<sup>2</sup> Laboratoire National des Champs Magnétiques Intenses, CNRS-UJF-UPS-INSA, 25, avenue des Martyrs, 38042 Grenoble, France

<sup>3</sup> Department of Physics, SUPA, Strathclyde University, G4 0NG Glasgow, UK

<sup>4</sup> Ural Federal University and Institute of Solid State Chemistry of RAS, Ekaterinburg, 620002, Russia

<sup>5</sup> Institute of Applied Physics and Mathematics, Faculty of Chemical Technology, University of Pardubice, Studentská 84, 532 10 Pardubice, Czech Republic

<sup>6</sup> Institute of Electronic Materials Technology, ul. Wolczynska 133, PL 01-919 Warsaw, Poland

*Scientific Reports*, 2016, 6, 19087

The Faraday effect is a representative magneto-optical phenomenon, resulting from the transfer of angular momentum between interacting light and matter in which time-reversal symmetry has been broken by an externally applied magnetic field. Here we report on the Faraday rotation induced in the prominent 3D topological insulator Bi<sub>2</sub>Se<sub>3</sub> due to bulk interband excitations. The origin of this non-resonant effect, extraordinarily strong among other non-magnetic materials, is traced back to the specific Dirac-type Hamiltonian for Bi<sub>2</sub>Se<sub>3</sub>, which implies that electrons and holes in this material closely resemble relativistic particles with a non-zero rest mass.

### Graphene FET Gigabit ON-OFF Keying Demodulator at 96 GHz

Habibpour O.<sup>1</sup>, He Z. S.<sup>1</sup>, Strupinski W.<sup>2</sup>, Rorsman N.<sup>1</sup>, Ciuk T.<sup>2</sup>, Ciepiewski P.<sup>2</sup>, Zirath H.<sup>1</sup>

<sup>1</sup> Chalmers, Microwave Elect. Lab., Dep. Microtechnol & Nanosci, S-41296 Gothenburg, Sweden

<sup>2</sup> Inst. Elect. Mat. Technol., 01-919 Warsaw, Poland

IEEE Electron Device Letters, 2016, 37, 3, 333 - 336

We demonstrate the demodulation of a multi-Gb/s ON-OFF keying (OOK) signal on a 96 GHz carrier by utilizing a 250-nm graphene field-effect transistor as a zero bias power detector. From the eye diagram, we can conclude that the devices can demodulate the OOK signals up to 4 Gb/s.

### Numerical simulations of epitaxial growth in MOVPE reactor as a tool for aluminum nitride growth optimization

Skibinski, J.<sup>1</sup>, Caban, P.<sup>2</sup>, Wejrzanowski T.<sup>1</sup>, Grybczuk M.<sup>1</sup>, Kurzydowski, K. J.<sup>1</sup>

<sup>1</sup> Warsaw Univ. Technol. Fac. Mat. Sci & Engn, Woloska 141, 02-507 Warsaw, Poland

<sup>2</sup> Inst. Elect. Mat. Technol., Wolczynska 133, 01-919 Warsaw, Poland

*Journal of Power Technologies*, 2016, 96, 2, 110 - 114

The present study concerns numerical simulations and experimental measurements on the influence of inlet gas mass flow rate on the growth rate of aluminum nitride crystals in Metalorganic Vapor Phase Epitaxy reactor model AIX-200/4RF-S. The aim of this study was to design the optimal process conditions for obtaining the most homogeneous product. Since there are many agents influencing reactions relating to crystal growth such as temperature, pressure, gas composition and reactor geometry, it is difficult to design an optimal process. Variations of process pressure and hydrogen mass flow rates have been considered. Since it is impossible to experimentally determine the exact distribution of heat and mass transfer inside the reactor during crystal growth, detailed 3D modeling has been used to gain insight into the process conditions. Numerical simulations increase the understanding of the epitaxial process by calculating heat and mass transfer distribution during the growth of aluminum nitride crystals. Including chemical reactions in the numerical model enables the growth rate of the substrate to be calculated. The present approach has been applied to optimize homogeneity of AlN film thickness and its growth rate.

### Direct comparison of shot-to-shot noise performance of all normal dispersion and anomalous dispersion supercontinuum pumped with sub-picosecond pulse fiber-based laser

Klimczak M.<sup>1</sup>, Sobon G.<sup>2</sup>, Kasztelaniec R.<sup>1</sup>, Abramski K. M.<sup>2</sup>, Buczynski R.<sup>1,3</sup>

<sup>1</sup> Inst. Elect. Mat. Technol., Glass Dept., Wolczynska 133, 01-919 Warsaw, Poland

<sup>2</sup> Wrocław. Univ. Technol., Laser & Fiber Elect. Grp., Wybrzeze Wyspianskiego 27, 50-370 Wrocław, Poland

<sup>3</sup> Univ. Warsaw., Fac. Phys., Pasteura 7, 02-093 Warsaw, Poland

*Scientific Reports*, 2016, 6, 19284

Coherence of supercontinuum sources is critical for applications involving characterization of ultrafast or rarely occurring phenomena. With the demonstrated spectral coverage of supercontinuum extending from near-infrared to over 10  $\mu\text{m}$  in a single nonlinear fiber, there has been a clear push for the bandwidth rather than for attempting to optimize the dynamic properties of the generated spectrum. In this work we provide an experimental assessment of the shot-to-shot noise performance of supercontinuum generation in two types of soft glass photonic crystal fibers. Phase coherence and intensity fluctuations are compared for the cases of an anomalous dispersion-pumped fiber and an all-normal dispersion fiber. With the use of the dispersive Fourier transformation method, we demonstrate that a factor of 100 improvement in signal-to-noise ratio is achieved in the normal-dispersion over anomalous dispersion-pumped fiber for 390 fs long pump pulses. A double-clad design of the photonic lattice of the fiber is further postulated to enable a pump-related seeding mechanism of normal-dispersion supercontinuum broadening under sub-picosecond pumping, which is otherwise known for similar noise characteristics as modulation instability driven, soliton-based spectra.

### World-smallest fiber-GRIN lens system for optofluidic applications

Filipkowski A.<sup>1</sup>, Piechal B.<sup>1</sup>, Pysz D.<sup>1</sup>, Stepień R.<sup>1</sup>, Ciemek J.<sup>1,2</sup>, Waddie A.<sup>3</sup>, Klimczak M.<sup>1</sup>, Stafiej P.<sup>4</sup>, Taghizadeh M. R.<sup>3</sup>, Buczyński R.<sup>1,2,3</sup>

<sup>1</sup> Department of Glass, Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

<sup>2</sup> Faculty of Physics, University of Warsaw, Pasteura 7, 02-093, Warsaw, Poland

<sup>3</sup> Department of Physics, School of Engineering and Physical Sciences, Heriot-Watt University, Scottish Universities Physics Alliance, Edinburgh, EH14 4AS, UK

<sup>4</sup> Faculty of Physics, Nicolaus Copernicus University, Grudziadzka 5, 87-10 Toruń, Poland

*Photonics Letters of Poland*, 2016, 8, 2, 36 - 38

We have developed a new type of optical fiber probe which integrates a standard single mode fiber with a gradient index (GRIN) microlens. The system is perfectly suited for optofluidic sensor applications since the diameter of the lens module is exactly the same as that of the optical fiber. Moreover, the performance of the GRIN lens is not degraded by low contrast of the refractive index between the lens and the fluidic environment. The GRIN lens is made with a novel technology of nanostructured optics.

### In situ Raman spectroscopy of the graphene/water interface of a solution-gated field-effect transistor: electron-phonon coupling and spectroelectrochemistry

Binder J.<sup>1</sup>, Urban J. M.<sup>1,3</sup>, Stepniński R.<sup>1</sup>, Strupinski W.<sup>2</sup>, Wyszomolek A.<sup>1</sup>

<sup>1</sup> Faculty of Physics, University of Warsaw, Pasteura 5, 02-093 Warsaw, Poland

<sup>2</sup> Institute of Electronic Materials Technology, Wólczyńska 133, 01-919 Warsaw, Poland

*Nanotechnology*, 2016, 27, 4

We present a novel measurement approach which combines the electrical characterization of solution-gated field-effect transistors based on epitaxial bilayer graphene on 4H-SiC (0001) with simultaneous Raman spectroscopy. By changing the gate voltage, we observed Raman signatures related to the resonant electron-phonon coupling. An analysis of these Raman bands enabled the extraction of the geometrical capacitance of the system and an accurate calculation of the Fermi levels for bilayer graphene. An intentional application of higher gate voltages allowed us to trigger electrochemical reactions, which we followed in situ by Raman spectroscopy. The reactions showed a partially reversible character, as indicated by an emergence/disappearance of peaks assigned to C-H and Si-H vibration modes as well as an increase/decrease of the defect-related Raman D band intensity. Our setup provides a highly interesting platform for future spectroelectrochemical research on electrically-induced sorption processes of graphene on the micrometer scale.

### Graphene growth on Ge(100)/Si(100) substrates by CVD method

Pasternak I.<sup>1</sup>, Wesolowski M.<sup>1</sup>, Jozwik I.<sup>1</sup>, Lukosius M.<sup>2</sup>, Lupina G.<sup>2</sup>, Dabrowski P.<sup>3</sup>, Baranowski J. M.<sup>1</sup>, Strupinski W.<sup>1</sup>

<sup>1</sup> Institute of Electronic Materials Technology, Wolczyńska 133, 01-919 Warsaw, Poland

<sup>2</sup> IHP, Im Technologiepark 25, 15236 Frankfurt (Oder), Germany

<sup>3</sup> Department of Solid States Physics, University of Lodz, Pomorska 149/153, Lodz, 90-236, Poland

*Scientific Reports*, 2016, 6, 21773

The successful integration of graphene into microelectronic devices is strongly dependent on the availability of direct deposition processes, which can provide uniform, large area and high quality graphene on nonmetallic substrates. As of today the dominant technology is based on Si and obtaining graphene with Si is treated as the most advantageous solution. However, the formation of carbide during the growth process makes manufacturing graphene on Si wafers extremely challenging. To overcome these difficulties and reach the set goals, we proposed growth of high quality graphene layers by the CVD method on Ge(100)/Si(100) wafers. In addition, a stochastic model was applied in order to describe the graphene growth process on the Ge(100)/Si(100) substrate and to determine the direction of further processes. As a result, high quality graphene was grown, which was proved by Raman

spectroscopy results, showing uniform monolayer films with FWHM of the 2D band of  $32 \text{ cm}^{-1}$ .

### Decrease of Nano-hardness at Ultra-low Indentation Depths in Copper Single Crystal

Kucharski S.<sup>1</sup>, Jarzabek D.<sup>1</sup>, Piątkowska A.<sup>2</sup>, Woźniak S.<sup>1</sup>

<sup>1</sup> Institute of Fundamental Technological Research, Warsaw, Poland

<sup>2</sup> Institute of Electronic Materials Technology, Warsaw, Poland

*Experimental Mechanics*, 2016, 56, 381 - 393

In the present study, we report a detailed investigation of the unusual size effect in single crystals. For the experiments we specified the hardness in single crystalline copper specimens with different orientations ((001), (011) and (111)) using Oliver-Pharr method. Our results indicates that with decreasing load, after the value of the hardness reached its maximum, it starts to decrease for very small indentation depths (<150 nm). For the sake of accuracy of hardness determination we have developed two AFM-based methods to evaluate contact area between tip and indented material. The proposed exact measurement of the contact area, which includes the effect of pile-up and sink-in patterns, can partially explain the strange behaviour, however, the decrease of hardness at low loads is still observed. At higher loads range the specified hardness is practically constant.

### Microstructure and Thermoelectric Properties of Bulk Cobalt Antimonide (CoSb<sub>3</sub>) Skutterudites Obtained by Pulse Plasma Sintering

Kruszewski M. J.<sup>1</sup>, Zybala R.<sup>1</sup>, Ciupiński Ł.<sup>1</sup>, Chmielewski M.<sup>2</sup>, Adamczyk-Cieślak B.<sup>1</sup>, Michalski A.<sup>1</sup>, Rajska M.<sup>3</sup>, Kurzydłowski K. J.<sup>1</sup>

<sup>1</sup> Faculty of Materials Science and Engineering Warsaw University of Technology, Warsaw Poland

<sup>2</sup> Institute of Electronic Materials Technology Warsaw Poland

<sup>3</sup> Faculty of Materials Science and Ceramics AGH University of Science and Technology, Krakow, Poland

*Journal of Electronic Materials*, 2016, 45, 3, 1369 - 1376

The use of the pulse plasma sintering technique for CoSb<sub>3</sub> thermoelectric material consolidation is reported in this work. The influence of sintering temperature on the microstructure and material properties such as the Seebeck coefficient, electrical resistivity, and thermal conductivity has been investigated. It is shown that, for samples fabricated at 923 K and 973 K, there were no significant differences in the average grain size or final phase composition. In both cases, a fine-grained polycrystalline structure of the compacts with density nearly equal to the theoretical value was achieved. Both samples were composed almost uniquely of CoSb<sub>3</sub> phase.

The measured thermoelectric parameters such as the Seebeck coefficient, electrical, and thermal conductivity showed similar dependence on temperature. For both samples, the Seebeck coefficient was negative at room temperature and showed a transition from n- to p-type conduction over the temperature range of 400 K to 460 K. The measured minimum thermal conductivity values,  $4 \text{ W m}^{-1} \text{ K}^{-1}$  to  $5 \text{ W m}^{-1} \text{ K}^{-1}$  at 723 K, are typical for undoped bulk CoSb<sub>3</sub>. A maximum ZT value of 0.08 at 623 K was obtained for the sample consolidated at 923 K for 5 min. The results of this work are very promising from the point of view of use of pulse plasma sintering as an alternative method for fabrication of a broad range of thermoelectric materials in the future.

### Computationally-efficient FDTD modeling of supercontinuum generation in photonic crystal fibers

Karpisz T.<sup>1</sup>, Salski B.<sup>1</sup>, Buczynski R.<sup>2,3</sup>, Kopyt P.<sup>1</sup>, Pancewicz A.<sup>1</sup>

<sup>1</sup> Institute of Radioelectronics and Multimedia Technology Warsaw University of Technology, Warsaw, Poland

<sup>2</sup> Institute of Electronic Materials Technology, Warsaw, Poland

<sup>3</sup> Information Optics Group, Faculty of Physics Warsaw University, Warsaw, Poland

*Optical and Quantum Electronics*, 2016, 48, 175

It is shown in this paper that a finite-difference time-domain method can be successfully applied to rigorous electromagnetic analysis of supercontinuum generation in photonic crystal fibers. Large computational requirements of the method are alleviated by the use of a hybrid procedure where, at first, vector two-dimensional simulation is applied in order to determine mode properties of the fiber. Subsequently, one-dimensional simulation of a pulse propagating in a transmission line filled with effective material is performed. The parameters of the line take into account nonlinear characteristics of the filling material as well as the previously computed mode dispersion. It is depicted that the proposed novel hybrid approach opens the way for rigorous, yet, computationally-efficient modeling of third order nonlinear processes in optically long fibers. The example investigated in this paper shows very promising results as compared with experiments and approximate numerical simulations of a nonlinear Schrodinger equation performed with the aid of the split-step Fourier method.

### Stable topological insulators achieved using high energy electron beams

Zhao L.<sup>1</sup>, Konczykowski M.<sup>2</sup>, Deng H.<sup>1</sup>, Korzhovska I.<sup>1</sup>, Begliarbekov M.<sup>1</sup>, Chen Z.<sup>1</sup>, Papalazarou E.<sup>3</sup>, Marsi M.<sup>3</sup>, Perfetti L.<sup>2</sup>, Hruban A.<sup>4</sup>, Wołoś A.<sup>5,6</sup>, Krusin-Elbaum L.<sup>1</sup>

<sup>1</sup> Department of Physics, The City College of New York, CUNY, New York, New York 10031, USA.

<sup>2</sup> Laboratoire des Solides Irradiés, École Polytechnique, CNRS, CEA, Université Paris-Saclay, 91128 Palaiseau cedex, France

<sup>3</sup> Laboratoire de Physique des Solides, CNRS, Université Paris-Saclay, Université Paris-Sud, 91405 Orsay, France.

<sup>4</sup> Institute of Electronic Materials Technology, 01-919 Warsaw, Poland.

<sup>5</sup> Institute of Physics, Polish Academy of Sciences, 02-668 Warsaw, Poland.

<sup>6</sup> Faculty of Physics, University of Warsaw, 00-681 Warsaw, Poland.

*Nature Communications*, 2016, 7, 10957

Topological insulators are potentially transformative quantum solids with metallic surface states which have Dirac band structure and are immune to disorder. Ubiquitous charged bulk defects, however, pull the Fermi energy into the bulk bands, denying access to surface charge transport. Here we demonstrate that irradiation with swift (~2.5 MeV energy) electron beams allows to compensate these defects, bring the Fermi level back into the bulk gap and reach the charge neutrality point (CNP). Controlling the beam fluence, we tune bulk conductivity from p- (hole-like) to n-type (electron-like), crossing the Dirac point and back, while preserving the Dirac energy dispersion. The CNP conductance has a two-dimensional character on the order of ten conductance quanta and reveals, both in Bi<sub>2</sub>Te<sub>3</sub> and Bi<sub>2</sub>Se<sub>3</sub>, the presence of only two quantum channels corresponding to two topological surfaces. The intrinsic quantum transport of the topological states is accessible disregarding the bulk size.

## Thulium Oxide Nanopowders Obtained by Precipitation

Sidorowicz A.<sup>1,2</sup>, Wajler A.<sup>1</sup>, Weglarz H.<sup>1</sup>, Jach K.<sup>1</sup>, Orłowski K.<sup>1</sup>, Olszyna A.<sup>2</sup>

<sup>1</sup> Institute of Electronic Materials Technology, Wolczynska 133, Warsaw 01-919, Poland

<sup>2</sup> Warsaw University of Technology, Wołoska 141, Warsaw 02-507, Poland

*Int. J. Appl. Ceram. Technol.*, 2016, 13, 2, 302 - 307

The aim of this work was to investigate the influence of precipitation parameters on the morphology of obtained thulium oxide powders. Tm<sub>2</sub>O<sub>3</sub> precursor powders were synthesized by precipitation method using 0.1 - 0.25 M water solutions of thulium nitrate and 1.5 M ammonium hydrogen carbonate water solution as a precipitation agent. The processes were conducted at different temperatures (25 - 50 degrees C). The result showed that the morphology of the obtained thulium oxide (Tm<sub>2</sub>O<sub>3</sub>) powders depends both on the molar concentration of thulium nitrate and the temperature of precipitation. Small, round, loosely agglomerated Tm<sub>2</sub>O<sub>3</sub> nanoparticles were obtained after air calcination of precursor

precipitated at room temperature with the use of 0.1 M thulium nitrate solution.

## Metal-ceramic functionally graded materials - manufacturing, characterization, application

Chmielewski M.<sup>1</sup>, Pietrzak K.<sup>1</sup>

<sup>1</sup> Institute of Electronic Materials Technology, Wolczynska 133, Warsaw 01-919, Poland

*Bulletin of the Polish Academy of Sciences Technical Sciences*, 2016, 64, 1, 151 - 160

Functionally graded materials (FGMs) belong to a new, continuously developing group of materials, finding application in various branches of industry. The idea of freely designing their construction profile, restricted only by the available manufacturing techniques, enables obtaining materials with composition and structure gradients having unprecedented properties. In this paper, selected results of works carried out by the authors and relating to the application of the developed metal-ceramic composites were presented in order to manufacture functionally graded materials for target purposes. Gradient structures with various construction profiles that can play different roles were produced on the basis on the following material pairs: Cr-Al<sub>2</sub>O<sub>3</sub>, NiAl-Al<sub>2</sub>O<sub>3</sub> and Cu-AlN. Manufacturing conditions, microstructure characteristics and selected properties, crucial from the point of view of future applications, were presented.

## Performance analysis of thermally bonded Er<sup>3+</sup>, Yb<sup>3+</sup>:glass/Co<sup>2+</sup>:MgAl<sub>2</sub>O<sub>4</sub> microchip lasers

Mlynczak J.<sup>1</sup>, Belghachem N.<sup>1</sup>, Kopczynski K.<sup>1</sup>, Kisielewski J.<sup>2</sup>, Stepień R.<sup>2</sup>, Wychowaniec M.<sup>3</sup>, Galas J.<sup>3</sup>, Litwin D.<sup>3</sup>, Czyzewski A.<sup>3</sup>

<sup>1</sup> Institute of Optoelectronics Military University of Technology, Warsaw, Poland

<sup>2</sup> Institute of Electronic Materials Technology, Warsaw, Poland

<sup>3</sup> Maksymilian Pluta Institute of Applied Optics, Warsaw, Poland

*Optical and Quantum Electronics*, 2016, 48, 247

The new glass as well as Co<sup>2+</sup>:MgAl<sub>2</sub>O<sub>4</sub> saturable absorber synthesis, especially developed for thermal bonding, was described. The procedure of thermal bonding was presented. Generation parameters of continuous wave operation at 1.5 μm wavelength were shown. The threshold below 180 mW and slope efficiency over 10 % was reached. Pulse generation in thermally bonded and unbonded as well as monolithic Er<sup>3+</sup>, Yb<sup>3+</sup>:glass/Co<sup>2+</sup>:MgAl<sub>2</sub>O<sub>4</sub> microchip lasers was compared. The peak power above 10 kW with pulse energy above 32 μJ and pulse width 3.2 ns was achieved.

## Wskazówki dla autorów

Redakcja wydawnictwa **Materiały Elektroniczne** prosi autorów o nadsyłanie zamówionych artykułów pocztą elektroniczną, pod adres [ointe@itme.edu.pl](mailto:ointe@itme.edu.pl) lub na nośniku magnetycznym, według następujących specyfikacji:

### Tekst

- a) Treść artykułu powinna być dostarczona w plikach o rozszerzeniu obsługiwanym przez program Word (najlepiej DOC i DOCX). Tekst powinien być pisany w sposób ciągły, podzielony na kolejno ponumerowane, zawierające tytuły, rozdziały. Oznaczenia zmiennych należy pisać czcionką pochyłą (kursywą). W tekście powinny być zaznaczone miejsca, w których mają znajdować się materiały ilustracyjne, jednak same grafiki powinny być umieszczone poza nim w oddzielnych plikach (patrz punkt 4).
- b) Podpisy do rysunków w języku polskim i angielskim, również winny być zapisane w oddzielnym pliku.
- c) Na pierwszej stronie artykułu powinny znajdować się następujące elementy: imię i nazwisko autora, tytuł naukowy, nazwa miejsca pracy, adres pocztowy, e-mail, tytuł artykułu zarówno w języku polskim jak i angielskim.

### Streszczenie

- a) Do artykułu należy dołączyć streszczenie w języku polskim i angielskim. Każde z nich nie powinno przekraczać 200 słów.
- b) Należy także dodać słowa kluczowe zarówno w języku polskim jak i angielskim.

### Bibliografia

- a) Pozycje bibliograficzne należy podawać w nawiasach kwadratowych w kolejności ich występowania.
- b) Sposoby sporządzania opisów bibliograficznych:

- Opis bibliograficzny całej książki:

Autor: Tytuł. Numer wydania. Miejsce wydania: Nazwa wydawca, Rok wydania, ISBN.

- Opis bibliograficzny pracy zbiorowej pod redakcją:

Tytuł. Pod red. (nazwiska redaktorów): Numer wydania. Miejsce wydania: Nazwa wydawca, Rok wydania, ISBN.

- Opis bibliograficzny fragmentu (rozdziału) książki, (gdy cała książka jest tego samego autorstwa):

Autor: Tytuł książki. Numer wydania. Miejsce wydania: Nazwa wydawca, Rok wydania, ISBN. Tytuł fragmentu, Strony rozdziału.

- Opis bibliograficzny fragmentu (rozdziału) książki z pracy zbiorowej:

Autor: Tytuł fragmentu. W: Tytuł książki. Miejsce wydania: Nazwa wydawca, Rok wydania, ISBN.

- Opis bibliograficzny artykułu z czasopisma:

Autor: Tytuł artykułu . „Tytuł czasopisma” Rok, Wolumin, Numer, Strony.

- Opis artykułu w czasopiśmie internetowym:

Autor: Tytuł artykułu [on line], Rok, Wolumin, Numer [dostęp – data] Strony, Adres w Internecie. ISSN

- Strona WWW:

Autor: Tytuł [on line]. Miejsce wydania: Instytucja sprawcza [dostęp – data], Adres w internecie.

### Elementy graficzne

- a) Każdy materiał ilustracyjny powinien być zapisany w oddzielnym pliku (PCX, TIF, BMP, WFM, WPG, JPG) o rozdzielczości nie mniejszej niż 150 dpi.
- b) W przypadku materiałów ilustracyjnych niebędących oryginalnym dorobkiem autora/ów należy zacytować ich źródło, umieszczając je w bibliografii.

### Wzory

- a) Wzory należy numerować kolejno cyframi arabskimi
- b) Zmienne należy oznaczyć czcionką pochyłą.
- c) W przypadku wzorów niebędących oryginalnym dorobkiem autora/ów należy zacytować ich źródło, umieszczając je w bibliografii.

**Autora obowiązuje wykonanie korekty autorskiej.**



# INSTYTUT TECHNOLOGII MATERIAŁÓW ELEKTRONICZNYCH

ul. Wólczyńska 133, 01-919 Warszawa

tel. - dyrektor: (+48 22) 639 58 05  
e-mail: [itme@itme.edu.pl](mailto:itme@itme.edu.pl)

tel.: (+48 22) 835 30 41  
[www.itme.edu.pl](http://www.itme.edu.pl)

Instytut Technologii Materiałów Elektronicznych jest wiodącym polskim ośrodkiem prowadzącym badania naukowe oraz prace badawczo-rozwojowe w zakresie fizyki ciała stałego, projektowania i technologii nowoczesnych materiałów, struktur i podzespołów dla mikro- i nanoelektroniki, fotoniki i inżynierii.

Badania te dotyczą następujących grup materiałów i ich zastosowań w postaci podzespołów:

- **materiały nowej generacji:** grafen, metamateriały, materiały samoorganizujące się i gradientowe, nanokryształy tlenkowe w różnych matrycach (szkło, tworzywa sztuczna);

- **materiały półprzewodnikowe i ich zastosowania:**

- **monokryształy** hodowane metodą Czochralskiego Si, GaAs, GaP, GaSb, InAs, InSb, InP i transportu z fazy gazowej SiC, o średnicach do 10 cm;

- **warstwy epitaksjalne** półprzewodnikowe uzyskiwane za pomocą metod CVO i MOCVO z Si, SiC, GaN, AlN, InN, GaAs, GaP, GaSb, InP, InSb oraz opartych o nie związków potrójnych i poczwórnych;

- **podzespoły** dla elektroniki i fotoniki: diody Schottky'ego, tranzystory FET i HEMT, lasery, fotodetektory, IR i UV;

- **materiały tlenkowe i ich zastosowania:**

- **monokryształy**, YAG domieszkowany: (Nd, Yb, Er, Pr, Ho, Tm, Cr), YVO: (Nd, Tm, Ho, Er, Pr) i podwójnie domieszkowany: (Ho + Yb, Er + Yb), GdVO<sub>4</sub>: (Er, Tm); LuVO<sub>4</sub>: (Er, Tm); GdCoB: (Nd, Yb) dla zastosowań laserowych; kwarc, LiNbO<sub>3</sub>, LiTaO<sub>3</sub>, SeBa<sub>(1-x)</sub>, Nb<sub>2</sub>O<sub>6</sub> dla zastosowań elektrooptycznych i piezoelektrycznych; CaF<sub>2</sub>, BaF<sub>2</sub>, jako materiały przezroczyste; Ca<sub>4</sub>GdO(BO)<sub>3</sub> jako materiał nieliniowy oraz NdGaO<sub>3</sub>, SrLaGaO<sub>4</sub>, SrLaAlO<sub>4</sub>, jako materiały podłożowe dla osadzania warstw nadprzewodników wysokotemperaturowych;

- **szkła** o zadanych charakterystykach spektralnych i szkła aktywne;

- **ceramiki** (Al<sub>2</sub>O<sub>3</sub>, Y<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>), ceramiki przezroczyste i aktywne;

- **Warstwy epitaksjalne** YAG: Nd, Cr dla zastosowań laserowych;

- **światłowodów** specjalne, fotoniczne, aktywne i obrazowody;

- **podzespoły dla elektroniki i fotoniki:** filtry i rezonatory z akustyczną falą powierzchniową; soczewki dyfrakcyjne, maski chromowe do fotolitografii;

- **inne materiały dla elektroniki:**

- **kompozyty** metalowo-ceramiczne, kompozyty metalowe;

- **złącza** zaawansowanych materiałów ceramicznych (Si<sub>3</sub>N<sub>4</sub>, AlN), kompozytów ceramiczno-metalowych i ceramik z metalami;

- **metale czyste** (Ga, In, Al, Cu, Zn, Ag, Sb);

- **pasty** do układów hybrydowych;

- **materiały** dla jonowych ogniw litowych, ogniw paliwowych i kondensatorów.

Instytut prowadzi również badania i wykonuje usługi w zakresie:

- **innych technologii HI-TECH:** fotolitografia, elektronolitografia, osadzanie cienkich warstw, trawienie, obróbka termiczna;

- **charakteryzacji materiałów:** spektrometria mas i Mössbauera, elektronowy rezonans paramagnetyczny (EPR), rozpraszanie wsteczne Rutheforda (RBS), absorbcja atomowa, wysokorozdzielcza dyfrakcja rentgenowska, spektroskopia optyczna i w podczerwieni (FTIR), pomiary widm promieniowania, fotoluminescencja, mikroskopia optyczna i skaningowa mikroskopia elektronowa i sił atomowych (AFM); spektroskopia głębokich poziomów: pojemnościowa (DLTS) i fotoprądowa (PITS), pomiary impedancyjne i szumów, temperaturowa analiza fazowa, pomiary dyfuzyjności ciepła.