R E V I E W

in a competition for the academic position of "professor" at IEES-BAS

in professional field 4.1 Physical sciences,

scientific specialty: “Physics of the Condensed Matter”,

publiched in Government newspaper of Bulgaria № 26/01.04.2022

Candidate: **Assoc. prof., PhD. Lilyana Petrova Kolaklieva- Central Laboratoryof Applied Physics (CLAP–Plovdiv), BAS**

Reviewer: **Prof. D.Sc. DORIANA IVANOVA MALINOVSKA, prof. emeritus at CL SENES-BAN**

The only candidate in the competition, **assoc. prof., Ph.D. Lilyana Petrova Kolaklieva**, is graduated from the Sofia University “St. Kliment Ohridsky”-Sofia in 1980 and has obtained the qualification of "physicist”. In 2000 she had defended Ph D thesis: “Creation and study of temperature stable ohmic and schottky contacts for high power SiC devices" and had been awarded the scientific and educational degree "PhD". In 1980, she started working as a physicist at the Laboratory of Applied Physics - BAS (now the Central Laboratory of Applied Physics – Plovdiv, BAS), where from 1987 to 2004, he held the following academic positions: assistant III degree, II degree, I degree and associate professor -II degree (associate professor), after holding relevant competitions, where she is currently an associate professor and is acting director of CLAP–Plovdiv, BAS. She specialized in several international research institutions in the USSR, in Greece and in Sweden.

1. **General description of the presented materials -** monographs, articles and reports, textbooks, certificates and patents, articles and reports, etc. (classification by subject); reduction due to matching etc. with justification).

The activities of **assoc. prof., Ph.D. Lilyana Petrova Kolaklieva** are related to scientifical and applied researchs mainly in two thematic fields: **I)** Formation and study of ohmic contacts for semiconductor devices operating at high temperatures and high power; **II)** Formation of nanostructured coatings for modification of metal surfaces and investigation of their properties in order to improve them for the purpose of their practical application.

Assoc. prof., Ph.D. Lilyana Petrova Kolaklieva participates in the competition with 22 publications in scientific journals, 2 book chapters and 3 author's certificates, which contain the results of performed research activities in the field of condensed matter physics - mainly fabrication of thin layers of semiconductor materials and metals and study of their composition, physical, electrical, structural, mechanical and thermal properties with a goal for their application in microelectronics and the metallurgy and mechanical engineering industry.

2.Publications before and after obtaining the scientific degree. Assessment of whether the candidate meets the minimum national requirements, the requirements in the LDAS (Low of Development of the Academic Staff in Bulgaria), the requirements of BAS and the requirements in the table attached to this document.

In the section "habilitation work" are included 8 of the publications presented in the competition published in refereed international journals with an impact factor and an impact range – 6 of them with IF and 2 of them – with SJR, which belong to the following categories according to the Regulations for the Application of

LDAS of RB: Q1 - 1 publication, Q2 - 6 publications and Q3 - 1 publication, and the sum of the points for this indicator is 160. Three of these publications are in the first formulated field of research, and the remaining 5 - in the second.

Outside the section “habilitation work” 14 publications are presented, which are in refereed international editions with an impact factor (4 publications with IF and 10- with SJR) in categories as follows: Q2 - 4 publications and Q3 - 4 publications and Q4 - 3 publications, 3 publications with SJR and 2 book chapters. 3 registered author's certificates are included to the publications outside the habilitation section, which demonstrate the scientific and applied contributions of the scientific research carried out by the candidate. 8 publications each. The sum of the points for this indicator (excluding the habilitation work) is 311.

The number of publications of the **assoc. Prof. Ph.D. Liliana Petrova Kolaklieva** before the awarding of the academic position of "associated professor" is 31. After her awarding of the academic position of "associated professor", the total number of publications with IF and SJR are 39, and 3 chapters in monographs and the 29 presentations at scientific forums. There are also 7 author certificates.

According to the National requirements of the Regulations for the implementation of the LDAS in the Republic of Bulgaria, there are the following points for the relevant groups:

i) Indicator A (theses for scientific and educational degree "PhD") - 50 points (with a minimum requirements -50 points);

ii) Indicator B (habilitation work) - 160 points (with a minimum requirements - 100 points);

iii) Indicator D (scientific publications) – 311 points (with a minimum requirement - 220 points);

iv) Indicator D (citations) – 592 points (for minimum requirements - 200 points);

v) Indicator E (participation in scientific projects) - 965 points (with a minimum requirement - 150 points).

The total number of points after obtaining the scientific degree "PhD" is 2028 (with a minimum requirement of - 670 points).

Assoc. prof., Ph.D. Lilyana Petrova Kolaklieva has a defended dissertation for awarding the scientific-educational degree "PhD" and was awarded the scientific title “scientific fellow- II degree” in 2004 (from HAC), which is equivalent to "associate professor" under the new LDAS of the Republic of Bulgaria.

Assoc. Prof. Ph.D. Lilyana Petrova Kolaklieva fully meets the minimum national requirements, the requirements of LDAS of Republic Bulgaria and the requirements of the National Assembly of IFTT-BAS for awarding the academic position "professor".

3. General characteristics of the candidate's scientific, scientific-applied and pedagogical activities;

The scientific research conducted by **Assoc. Prof. PhD. Lilyana Petrova Kolaklieva** in the first formuated scientific area is related to the development and optimization of technologies for depositing and formation of ohmic contacts to the NEMT structure AlGaN/GaN [A1.1, A 1.2]. Ti/Al contacts with low contact resistivity and stable at operating temperatures up to 400^0C have been created. Metal structures (Ti/Al)Ti/Al and (Ti/Al)MoAu have been also developed, and the influence of the formation temperature, the ratio of the elements on the values of the contact resistivity has been studied. An explanation of the formation mechanism and the ratio of metals in the near-contact and barrier layer (Ti or Mo) and in the contact structures (Ti/Al) is proposed. It has been found that with increasing in the forming temperature the value of the contact resistance increases, which is explained by increasing of the diffusion of elements, as a result Ti and Al reach the surface and are oxidized. It was found that a Mo-barrier layer is formed, which decrease the intensity of Au and Al diffusion [A1.3]. The influence of the ratio of (Ti/Al) and of the barrier layer on the morphology and composition of the surface was investigated [A1.2]. **For the first time**, a thermostability test was conducted in order to investigate the reliability of (Ti/Al) contacts to the NEMT structure AlGaN/GaN. It has been

established that the increase in Ti content and the formation of TiN, as well as the presence of Mo as a buffer layer in the contact structure, provides excellent temperature stability in the entire temperature range 25°C - 400°C [A1.2]. The influence of the Ti:Al ratio on the morphology and composition of low-resistance Au/Ti/Al contacts to 4H-pSiC was investigated and it was found that increased aluminum content determined a higher optimum formation temperature, 900 °C and 1000 °C, respectively for ratio 70%:30% and 30%:70%. This is associated with the diffusion of Al during annealing, which may limit the interactions at the interface with SiC [B1.3]. It has been found that annealing at the optimum forming temperature leads to increasing of the roughness and this effect is more pronounced in contacts with increased Al content [B1.3, B1.4]. Through XPS analysis, it was found that regardless of the Ti:Al ratio and the annealing temperature respectively, TiC is formed at the contact/SiC interface [B1.3, B1.4]. Investigation of the phase composition by TEM shows the presence of the binary compounds Au_2Ti and Al_3Ti in both contact structures, which contribute to charge transfer and thus improve the ohmic properties [B1.6]. Binary and ternary compounds that are formed in the contact layer upon annealing are found to facilitate carrier transfer across the contact. The Au/Ti/Al structure was applied as an ohmic contact in the developed p-i-n 4H-SiC diode, on the basis of which a high-speed modulator in the Q-region was created [B1.2]. A study of the processes in the Au/Pd/Ti/Pd/SiC system during annealing was carried out and the mechanisms of the formation of ohmic properties were explained, as well as their thermostability during heat treatment up to 700 ^{o}C in a nitrogen environment and at operating temperatures up to 450 ^{o}C at air. Annealing the contact at a temperature of 900 ^{o}C, which is optimal for achieving minimum contact resistance, initiates and activates physico-chemical processes leading to the formation of new compounds with a different distribution in the contact/interface/semiconductor system. It was found that at this temperature a partial dissociation of the SiC surface to silicon and carbon occurs at the Pd/SiC interface, resulting in the interface being displaced into the SiC bulk [B1.1]. It has been shown that the presence of Ti and Au in the contact composition strongly limits Pd agglomeration and improves the surface morphology. Mainly the contact surface consists of gold and palladium silicides. In the base region, the Pd/Si ratio suggests the presence of PdSi, while in the agglomeration region, Pd_2Si and unreacted Pd are detected [B1.5]. Al-free AlGaN/GaN HEMT structure contacts were developed **for the first time** in order to improve the surface morphology and edge sharpness of the contact after formation of ohmic properties. Due to the tendency of Al to form droplets at higher temperatures, the surface of the Al-containing contacts has high roughness and also degraded edge sharpness. Two contact structures Ti/M/Au and Ti/M/Ti/Au were developed, in which the hard-melting metals Mo, Ni and Pd are denoted by M [B1.7, B1.8]. Between the investigated contact structures not containing Al, the Ti/Mo/Ti/Au structure was determined as optimally combining good electrical (ρc= 8.8x10-6 $\Omega.cm^2$) and morphological properties (R_{ms}=16 nm, D = 72 nm). This contact is considered as an alternative to Al containing contacts.

Scientific researchs from the second research field is related to the study of the mechanical properties, structure, composition, oxidation and corrosion resistance of various nanocomposite coatings for high-temperature application in the metalworking industry TiAlSiN [A2.1, A2.2], CrAlSiN[A2.3], CrAlSiN-AlSiN with periodically modulated composition [A2.4]. Very good corrosion resistance of TiAlSiN has been established with potential for dry machining applications. This coating is introduced in industrial production and is applied to cutting metalworking tools and dies as is mentioned in the author's reference. Research on the gradient CrAlSiN coating has shown that it is resistant to oxidation at temperatures of 800-900°C and is suitable for application on high-speed steel tools that operate in this temperature range. **For the first time**, a CrAlSiN-AlSiN nanocomposite coating with periodically modulated composition was created, combining ultra-hardness and high resistance to elastic and plastic deformations. The mechanical properties, composition and structure of the developed coating are investigated and the relationship between them is discussed [A2.4]. The temperature resistance of the coating was investigated in the range 600^{o}C – 900^{o}C by prolonged heating in an Ar environment [A2.5]. It was found that the CrAlSiN-AlSiN coating with a periodically modulated composition retains the combination of superhardness with resistance to elastic stresses and plastic deformations up to 800 °C, indicating its potential for high-temperature applications. Experiments have been

conducted to investigate the influence of thermal treatment, immediately after application, on the mechanical properties of the gradient coating TiAlSiN [B 2.1]. The coating has been obtained by cathodic arc evaporation at a temperature of 500^{o}C. Immediately after its deposition in the chamber, it is annealed at 525°C under nitrogen. By studying of the mechanical properties, a substantial increase in the hardness of the coating has been found from 36 GPa to 48 GPa after annealing. Adhesion-cohesion properties have been also significantly improved. As a result of the improved mechanical and adhesion-cohesion properties, very good wear resistance of the TiAlSiN gradient coating was achieved at low (0.02 m/s, 42.46 rpm) and high (0.20 m/s, 424.63 rpm) friction speeds [B2.2]. An approach is proposed to improve the mechanical properties and wear resistance of Ti-based coatings, which are one of the most widely applied in the modern metalworking industry. A range of low-temperature (<200 ^{o}C) technologies for the deposition by unbalanced magnetron sputtering of multicomponent (triple and quadruple) transition metal nitrides (Ti, Cr, Al) has been developed and their mechanical properties, morphology, composition and structure have been investigated [B2.3 -B2.7]. It has been shown that stable structure and composition, as well as good adhesion, are achieved in the low-temperature production technology [B2.7]. The mechanical properties of an AlN coating obtained by pulsed laser deposition have been investigated, depending on the technological conditions [B2.8]. In this study, it has been found that depending on the technological parameters (deposition temperature, pulse frequency, reactive gas pressure), the hardness of the AlN layers varies in the range of 22 GPa – 30 GPa. The temperature dependence of the resistance to elastic and plastic deformations has been determined from the measured hardness and elasticity coefficient. The result demonstrate that AlN coatings deposited by laser deposition have a very good resistance to elastic deformation, which is practically independent of the deposition temperature [B2.8].

The main scientific and applied contributions are related to developments intended for innovative transfer in industry. These contributions are intellectual property protected by utility models. 3 author's certificates have been submitted. The composition and structure of a TiAlSiN [C2-1] multilayer nanocomposite coating has been proposed **for the first time**. The structure and composition are subject to protection, including the percentage content of each element of the TiAlSiN multilayer nanocomposite coating, as well as the sequence of application of the composite layers and their thickness. **For the first time**, a planetary mechanism has been proposed, providing continuous and uniform movement of devices intended for the uniform application of single- and multi-layer wear-resistant coatings [C2-2]. The subject of protection is the construction of the planetary mechanism. **Forthe first time,** a multilayer coating for dental instruments is offered, which is characterized by a high degree of hardness, elasticity and a low coefficient of friction [C2-3]. The subject of protection is the structure of the coating, the composition and thickness of each of the constituent layers and their sequence when building its structure.

Assoc. prof. Ph.D. Lilyana Petrova Kolaklieva has participated in the period 1995-2007 in research activities in 6 international research projects and contracts, financed by EC programs COPENUCUS, NATO-Science for peace, INTAS, INCO, PHARE-BG and with BB Corporate Research-Sweden.

In the period 1993-2022 she has participated in a significant number of national research projects and contracts with the National Research Institute, with the Ministry of Economy under the program "Strengthening the competitiveness of the Bulgarian economy", with the National Investment Fund. She also has participated in the activities under bilateral innovative contracts with "SolTech" EOOD, "Milko Angelov Consult" EOOD and "Nanotech Group" EOOD. The research activities under these contracts are in the field of scientific and applied research of the applicant related to the development of technologies for creating ohmic contacts to semiconductor materials and coatings on metal surfaces.

4. Attention should also be paid to the pedagogical activity of the candidate from the beginning of his career: lectures, exercises, writing textbooks and study aids, supervision of doctoral students, specialists and graduates students

Assoc. prof. Ph.D. Lilyana P. Kolaklieva has also a pedagogical activity - she has been the supervisor of two doctoral students in the Central Laboratory of Applied Physics - BAS, which have been dismissed with the right of defense in 2013 and in 2017, and she is now the supervisor of one PhD student since 2020.

5. Basic scientific and scientific-applied contributions (for each contribution it is indicated where it is contained), to meaningfully analyze the scientific achievements of the candidate, clearly stating the nature of the scientific contributions:

- proving with new means of essential new aspects of already existing scientific fields, problems, theories, hypotheses; The mechanical properties of an AlN coating obtained by pulsed laser deposition have been investigated, depending on the technological conditions [B2.8]. AlN coatings deposited by laser deposition have been shown to have very good resistance to elastic deformation, which is practically independent of the deposition temperature.

- creation of new methods, constructions, technologies; A technology for applying and forming ohmic contacts to an AlGaN/GaN HEMT structure has been developed and optimized [A1.1, A 1.2]. **For the first time,** the reliability of (Ti/Al) contacts to AlGaN/GaN HEMT structures was investigated through a thermostability test [A 1.2]. **For the first time**, a CrAlSiN-AlSiN nanocomposite coating with periodically modulated composition has been created, combining ultra-hardness and high resistance to elastic and plastic deformations [A 2.4]. Al-free AlGaN/GaN HEMT structure contacts have been developed **for the first time** in order to improve the surface morphology and edge sharpness of the contact after formation of ohmic properties [B1.7, B1.8]. A range of low-temperature (<200 ^{o}C) technologies for the deposition by unbalanced magnetron sputtering of multicomponent (triple and quadruple) transition metal nitrides (Ti, Cr, Al) has been developed and their mechanical properties, morphology, composition and structure have been investigated [B2.3 -B2.7]. Composition and structure of TiAlSiN [C2-1] multilayer nanocomposite coating have been proposed **for the first time**. **For the first time**, a planetary mechanism has been proposed, providing continuous and uniform movement of devices designed for the uniform application of single- and multi-layer wear-resistant coatings. **For the first time**, a multi-layer coating for dental instruments has been proposed, which is characterized by a high degree of hardness, elasticity and a low coefficient of friction [C2.3].

-formulation (justification) of a new hypothesis; **A new hypothesis** has been proposed that explains the mechanism of formation of ohmic properties and the role of the ratio (Ti/Al) in the first contact layer, as well as of the barrier layer (Ti or Mo) in (TiAl) contact structures [A1.1,A1.3] . **A new hypothesis** has been proposed explaining the mechanisms of formation of ohmic properties in the Au/Pd/Ti/Pd/SiC system during annealing, as well as their thermostability during thermal treatment up to 700 ^{o}C in a nitrogen environment and at operating temperatures up to 450 ^{o}C in air [B1. 1].

- implementation contributions: methods, constructs, preparations, schemes, etc. The developed TiAlSiN [B2.1] coating has been used in industrial production and has been applied to cutting metalworking tools and dies for dry machining.

6. Reflection of the candidate's scientific publications in our and foreign literature (according to his data):

Assoc. Prof. Ph.D. Lilyana Petrova Kolaklieva, has **a total Hirsch index h = 9** (without self-citation by the authors). The number of the publications with an impact factor (IF) with which she participated in the announced competition are 6 and a total **IF is 10,867.**

The published results of the overall research activity of **Assoc. Prof. Dr. Lilyana Petrova Kolaklieva** have been widely reflected in the international scientific community. A total of 339 citations from independent authors of 60 publications are observed.

296 citations were noted in publications by independent foreign authors of 28 of the applicant's publications with which she participated in the competition, the citations being mainly in IF journals,

published books, Proceedings of international conferences and dissertations: 1 publication is cited 90 times, 1 -19 - times, 1 - 16 times, 2 - 11 times, 2 - 10 times, 4 - 9 times, 3 -8 times, etc.

-type of citations (it is in the applicant's interest to submit copies of the essential citations, to assess their character).

A large number of citing publications has pointed the significance of the results obtained in the candidate's publications. E.g. in citation [39 of the Complete list of citations] *(Properties of Al ohmic contacts to n-type 4H-SiC employing a phosphorus-doped and crystallized amorphous-silicon interlayer, Hanafusa, H., Ohta, A., Ashihara, R., Maruyama, K., Mizuno, T., Hayashi, S., Murakami, H., Higashi, S., MATERIALS SCIENCE FORUM, Volume 778-780, 2014, Pages 649-652. https://doi.org/10.4028/www.scientific.net/MSF.778-780.649)* pointed the result in the published work [1 of the candidate's total list of publications] on the observed C aggregation process on the NiSi surface as a result of the accumulation of C atoms during the annealing process, which is critical for the ohmic contact properties to NiSi. In the publication [243 of the Complete list of citations*] „Anisotropic hole drift velocity in 4H-SiC, Vasconcelos, Jackelinne L.; Rodrigues, Cloves G.; Luzzi, Roberto, MATERIALS SCIENCE AND ENGINEERING B-ADVANCED FUNCTIONAL SOLID-STATE MATERIALS, Volume: 249, Article Number: 114426, Published: OCT 2019. DOI: 10.1016/j.mseb.2019.114426*“ is pointed that the authors' results are comparable to those published in the article by **Assoc. Prof. Dr. Lilyana Petrova Kolaklieva** *„L. Kasamakova-Kolaklieva, L. Storasta, I.G. Ivanov, B. Magnuson, S. Contreras, Consejo, J.Perno, M. Zielinski and E.Janzen, Temperature-dependent Hall effect measurements in low-compensated p-type 4H-SiC, Materials Science Forum, 457-460, 677-680 (2004). DOI:https://doi.org/10.4028/www.scientific.net/MSF.457-460.67“.*

In the citing publication [274 of the Complete list of citations] *(„Modelling temperature dependence on AlGaN/GaN power HEMT device characteristics, Wang, Y.-H., Liang, Y.C., Samudra, G.S., Chang, T.-F., Huang, C.-F., Yuan, L., Lo, G.-Q., SEMICONDUCTOR SCIENCE AND TECHNOLOGY, Volume 28, Issue 12, December 2013, Article number 125010. DOI:10.1088/0268-1242/28/12/125010“)* the authors have been used the proposed hypothesis in the in publication [39] *(L. Kolaklieva, R. Kakanakov, Ohmic Contacts for High Power and High Temperature Microelectronics, Chapter 18 in "Microelectronic and Mechenical Systems“edit. Kenichi Takahata, ISBN 978-953-307-027-8, p. 293-318, 2009)* to explain the results by the thermionic mechanism, which is enhanced at higher temperatures during carrier transport across the metal-semiconductor transition.

In the citing publication № 332 [from the Complete list of citations] *(„The Effect of Substrate Treatment on the Properties of TiAlSiYN/CrN Nanocomposite Coatings, Alexander Pogrebnjak, Vladimir Buranich, Volodymyr Ivashchenko, Lazat Baimoldanova, Krzysztof Rokosz, Steinar Raaen, Pawel Zukowski, Marek Opielak, Bauyrzhan Rakhadilov, Vyacheslav Beresnev, Nazgul Erdybaeva, Surfaces and Interfaces (2022), doi: https://doi.org/10.1016/j.surfin.2022.101902.“),* it is noted that the determined H3/E2 ratio in publication No. 60 of **Assoc. Prof. Dr. Lilyana Petrova Kolaklieva** *(„L. Kolaklieva, R. Kakanakov, P. Stefanov, D. Kovacheva, G. Atanasova, S. Russev, V. Chitanov, T. Cholakova, C. Bahchedjiev, Mechanical and Structural Properties of Nanocomposite CrAlSiN–AlSiN Coating with Periodically Modulated Composition, Coatings 2020, 10, 41; doi:10.3390/coatings10010041“),* as an indicator of the resistance of materials to plastic deformation to predict the formation of a shear band under local deformation in the material, is an important indicator for this assessment.

7. In the case of collective publications, the candidate's contribution should be separated, and in case of a plagiarism report, an opinion should be given regarding the presence or absence of plagiarism (LDAS in Bulgaria, art. 10 (2)).

The opinions of co-authors in the publications of **Assoc. Prof. Dr. Lilyana Petrova Kolaklieva** are presented regarding her significant contributions to the conducted research, which are published in the

corresponding articles. There is no presence of plagiarism, according to LDAS of the Republic of Bulgaria, art. 10 (2)).

8. Critical notes of the reviewer on the presented works, including the candidate's literary awareness.

I have no critical notes

9. Personal impressions of the reviewer about the candidate and other data not specified in the previous points.

10. Motivated and clearly formulated conclusion.

Research activity of **Assoc. Prof. Ph.D. Lilyana Petrova Kolaklieva** is mainly related to preparation and research in the field of materials science, preparation of thin layers of semiconductor materials and study of their composition, physical, electrical, structural, mechanical and thermal properties with potential for their application in microelectronics and the metalworking industry and mechanical engineering. The contributions in the presented scientific publications are important both in their fundamental nature and in terms of the application of the materials.

The presented materials for the competition meet entirely the requirements under Art. 29, para. (1), item 5 of the LDAS in RB and the recommended requirements for the academic position "professor" and defined in Appendix No 1 of the Regulations for the terms and conditions for acquiring scientific degrees and for occupying academic positions at ISSP-BAS.

On the basis of the presented scientific publications and the research activity in the field of Condensed Matter Physics, I propose to the scientific jury of the competition to recommend to the Scientific council of the ISSP-BAS to vote the candidate in the competition, the **assoc.prof. Ph.D. Lilyana Petrova Kolaklieva** to obtain the academic position of "Professor", professional direction 4.1 "Physical sciences" (Scientific specialty "Physics of condensed matter").

15.07.2022 Reviewer: