R E V I E W

on a procedure for the occupation of the academic position "Professor" in the professional field 4.1. "Physical Sciences", specialty "Condensed Matter Physics" (Creation and investigation of thin layers for surface modification) according to the announcement of the Central Laboratory of Applied Physics (CLAP) - Bulgarian Academy of Sciences (BAS) in the State Gazette, issue 26/01.04.22 .

Candidate: Liliana Petrova Kolaklyeva, Ph.D., Assoc. Professor at CLAP - BAS

Reviewer: Dianka Dimitrova Nesheva-Slavova, Dr.Sci., Professor at Institute of Solid State Physics (ISSP)-BAS

General characteristics of the submitted materials*

Assoc. Prof. PhD Liliana Petrova Kolaklieva is the only candidate for this competition and has submitted all required materials for participation in the competition.

Assoc.Prof. L.Kolaklieva graduated from Faculty of Physics, Sofia University "St. Kliment Ohridski" in 1980 with a specialization in "Physics of semiconductors" and the same year she was appointed as a "physicist" in the Laboratory of Applied Physics - BAS. In 2000 she obtained her PhD degree on "Creation and research of temperature-stable ohmic and Schottky contacts for high-power devices from SiC", supervised by Prof. Jordan Kassabov. Assoc. Prof. Kolaklieva specialized at the Institute of Semiconductor Physics, SO AN, Russia (1986), at the Institute of Electronic Structures and Lasers in Crete, Greece (2000) with funding from the NATO-Science for Peace program and was of post-doctoral specialization in the Department of Materials Science at Linkoping University, Sweden (2002 - 2004). She has the skills to work with a variety of vacuum equipment for deposition of nanocomposites and multilayer coatings and with specialized equipment's for complex layer characterization. She has successively held the following academic positions: research assistant third degree at the Institute of Applied Physics (1987 - 1997), research assistant second (1997 - 2000) and first (2000 - 2004) degree at the Institute of Applied Physics - BAS. Since 2004, she has been an associate professor and head of the "Technology and characterization of thin layers" group at the CLAP. In the periods 2011-2012 and 2021-2022, she was the deputy director, and in the period 2012-2021 and at the moment - the director of CLAP.

Assoc. Prof. Kolaklieva is co-author of a total of 101 scientific publications that received 339 independent citations (h-index 9) and 7 patented utility models. She participated in the development of 11 scientific and applied technologies and in a large number of financed projects - 6 international projects under the Copernicus, NATO, INTAS, PHARE programs, 1 under program Development of the competitiveness of the Bulgarian economy, 2 under OP NOIR, 10 projects financed by the Nacional Science Fund, Bulgarian Ministry of Education and Science, Ministry of Economy, National Innovation Fund. She participates in bilateral research projects and works in collaboration with scientists from Russia, Sweden, Belarus and Greece. It should also be noted its participation in bilateral innovative contracts with companies ("SolTech" EOOD, "Milko Angelov Consult" EOOD, "Nanotech Group" EOOD, etc.), as well as in the organization of scientific forums and editing their materials for publication.

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* The indicators and quartiles cited below are defined in the Implementing Regulations of the Law on Development of the Academic Staff in the Republic of Bulgaria (ZRAS RB), Professional field 4.1, Tables 1 and 2

In the competition Assoc. Prof. Kolaklieva participated with the following materials: indicator *B4* – habilitation work including 8 papers published in the period 2009-2021, providing 160 points, the required minimum is 100 points; of them 1 paper is in quartile Q1, 6 - in Q2, 1 - in Q3; in 5 publications she is the first author and in the others she has a substantial contribution. All 16 publications presented in indicator *G7-8* were published after obtaining the PhD degree and the competition for "associate professor" and are not included in the NACID database, to cover the minimum national requirements for occupying position Assoc. Prof. Of them, 2 papers are chapters in books with the first author Assoc. Prof. Kolaklieva, 4 are in journals with Q2, 4 - in Q3, 3 - in Q4, 3 - in journals without impact factor and together with the three patented utility models in *G9* provide 311 points in indicator *G* at min. 220 points. In 6 of these publications, the candidate is the first author, in 3 - second and in 3 - third. She participated in the competition with a list of 296 independent citations of 58 publications (592 points according to *D11*, with min. 200). Highly cited are works numbered 1 (90 times), 6 (19 times) and 2 (15 times) in this list. Dr. Kolaklieva has provided many examples of substantial citations of her publications that convincingly demonstrate a good knowledge of her works and use of the reported results and proposed explanations. One example: in work by Wang, Y.-H., Liang, Y.C., Samudra, G.S., Chang, T.-F., Huang, C.-F., Yuan, L., Lo, G.-Q. , Semicond. Sci. Technol. 28, 125010 (2013) states “Although carrier transport across the contact is ideally dominated by the temperature-independent tunneling process, both contact resistance and contact resistivity are observed to decrease at higher temperatures. This can be explained by the involvement of a thermoelectronic mechanism, which is enhanced at higher temperature, during carrier transport through the metal-semiconductor transition [L. Kolaklieva, R. Kakanakov, Chapter 18 in "Micro Electronic and Mechanical Systems", Edit. Kenichi Takahata, p. 293-318 (2009)]. The assets presented in group *E* indicators (leader of the CLAP team in 2 centers under OP NOIR program and 1 INFRAMAT project, leader of 2 national projects, participation in 3 international and 2 national projects and the funds obtained for these projects) correspond to 965 points with min. 150 points. I would like to pay attention to the introduction of the Author's contributions. It concisely and competently outlines the two main thematic areas in which are the achievements of Dr. Kolaklieva, submitted for the competition. The actuality, the problems, the possible ways to solve them and the applicability of the results are indicated, and this convinces of the importance of the conducted research and the significance of the obtained results.

Assoc. Prof. Kolaklieva also has a significant pedagogical activity - she was the supervisor of four successfully graduated diploma students, of two PhD students, dismissed with the right of defense their thesis and currently supervises one PhD student.

The attainments with which Assoc. Prof. L. Kolaklieva participated in the competition far exceed the minimum national requirements for occupying the academic position of "professor", specified in the Regulations for ZRAS RB and in the related requirements of BAS and ISSP. They present her as an actively working scientist with high competence, serious scientific and applied contributions and pedagogical experience. Her achievements are well known and used by the international scientific community, and some of them are close to practical application.

General characteristics of the applicant's scientific and applied activities

The achievements of Assoc. Prof. Kolaklieva presented for the competition are in the field of metal and non-metal layers with complex composition and structure with focuses on two topic areas: (1) high-power and high-temperature microelectronics (incl. powerful high-voltage unipolar transistors and powerful bipolar diodes and transistors) and (2) hard and superhard coatings for effective surface modification of materials used in modern industry (metalworking, automotive, optical applications, etc.). In the first area, SiC and III-V nitrides, and in particular GaN, are of

great interest since they allow the development of devices with high breakdown electric fields, high thermal conductivity and electron mobility. The quality of the contacts to SiC and GaN is crytical for the good and stable operation of such devices, and the research conducted by Dr. Kolaklieva is related to this important problem of present interest. No less contemporary and interesting are the nanostructured hard and superhard coatings developed with her participation. They have improved mechanical and tribological properties, high temperature stability, corrosion and wear resistance and have potential for various applications.

The developed electrical contacts and hard nanostructured coatings are multilayered or multicomponent, and for each of them a technology with appropriate deposition parameters has been developed. Different methods of physical vapor deposition (thermal, electron beam and cathode arc evaporation, unbalanced magnetron sputtering, etc.) have been used. The latter two methods mentioned were chosen because they are used in industry and would facilitate the transfer of the developed hard coatings. For the complex characterization of the samples, a large set of modern techniques suitable for investigation of morphology, structure and composition of nanosized and nanostructured objects (TEM, SEM, AFM, EDX, XPS, Raman spectroscopy, etc.) have been applied together with techniques for studying electrical and thermal properties, hardness, etc.

Main scientific and applied contributions

The main contributions of Assoc. Prof. Kolaklieva are grouped in accordance with the above two topic areas, as in each area articles from indicators *B4* and *D7-9* are considered.

Topic area 1. Results of complex studies of surface morphology, chemical and phase composition, electrical and thermal properties of aluminum-based and Al-free multilayer ohmic contacts, built from nanolayers of different thicknesses, which were deposited on the GaN top layer of GaN/AlGaN/GaN heterostructures or p-type 4H-SiC layers were presented. Mechanisms for the formation of ohmic properties are proposed. Specifically:

1.1. It was found that the starting weight ratio of the first two Ti/Al layers (closest to the GaN layer) of Ti/Al/Ti(Mo)/Au contacts to GaN/AlGaN/GaN type affects the contact resistance, the temperature of annealing that produces ohmic contact with the lowest resistance, as well as the elemental distribution and interface chemistry of the annealed contacts. Replacement of the second titanium layer with Mo layer hinders the diffusion of Al to the contact surface and reduces its surface roughness, improves the sharpness of the contact edges and its thermal stability during operation. The ohmic properties of the contacts are related to the formation of $AlAu_2$ in the contact layer and TiN at the interface with GaN. (*B4*:A1-A3; *D8*:B1,B2; the cited article numbers correspond to the arrangement in Lists of scientific works, inventions, citations …)

1.2. It was found for Al/Ti/Au ohmic contacts to p-type 4H-SiC with different weight ratio of Al and Ti that it does not affect the lowest specific contact resistance value, but it affects the optimum annealing temperature at which this value is obtained, which means different elemental distribution and interface chemistry in the annealed contacts. (*D7*:B3,B5; *D8*:B2) Besides, p-i-n diodes based on 4H-SiC were made with Al/Ti/Au contact to the p-type 4H-SiC layer, which have very good characteristics. With such a diode a high-speed modulator was developed having low forward voltage drop at a current density of 100 A/cm^2, leakage current below 1 μA at a voltage of 1200 V, and operating speed of 5-7 ns. These parameters indicate that the modulator is very promising for Q-band applications. (*G7*:B2)

1.3. In order to achieve low roughness and good contact edge sharpness after heat treatment to form ohmic properties, an original development of Al-free contacts to GaN/AlGaN/GaN was made. It has been demonstrated that Ti/Mo/Ti/Au and Ti/Ni/Ti/Au contacts have very low resistance and good surface smoothness. By optimizing the content of Ti in the contact composition a much better sharpness of their edge than that of contacts based on Al was achieved. It is concluded that these contacts are a very good alternative to contacts with Al. (*D7*:B6; *D8*:B1).

1.4. Studies on several types of contacts based on Pd (Au/Pd, Au/Pd/Al and Au/Pd/Ti/Pd) show that palladium is suitable for making ohmic contacts with low resistance and good thermal properties to p-type 4H-SiC for submicron devices, but further research is needed to reduce the surface roughness. The appearance of ohmic properties upon annealing was found to be due to the formation of PdSi at the interface with SiC, leading to a strong reduction in the potential barrier height. (*D7*:B1,B4; *D8*:B2)

Topic area 2. Multicomponent nanocomposite coatings with very high hardness, high temperature stability, high wear and oxidation resistance, low friction coefficient have been developed, which are superior to currently used coatings of metal-cutting instruments, dies or press that are based on two or three component layers of transition metal nitrides. Specifically:

2.1. Two multilayer coatings targeting specific applications have been developed and each is patented as a utility model. The first is a multilayer gradient nanocomposite coating based on TiAlSiN for application in the metalworking industry. (*B4*:A4,A5; *D9*:C1)) It contains both a gradient nanolayer of TiAlSiN and a nanocomposite layer of TiAlN/SiN. The coating has a high hardness (40 GPa) related to the nanocomposite layer consisting of nanocrystals of TiN, AlN and (TiAl)N with sizes of 5-8 nm in an amorphous matrix of Si_3N_4 and good adhesion to the substrate, which is attributed to the presence of the gradient layer. It has a low friction coefficient, shows slight oxidation of the surface at 800 °C without significant changes in its structure and composition, it is resistant to high temperature and corrosion treatment. All these make it suitable for industrial application in dry metalworking. According to the applicant's information, this coating has already been implemented in cutting metalworking tools and dies. The second multilayer coating is intended for dental instruments. It consists of a buffer layer/intermediate gradient layer/finishing diamond-like layer. (*G9*:C3)

2.2. Gradient coatings based on Ti and Cr have been developed. The coating of TiAlSiN has a high wear resistance (*G7*:B7,B8) and that of CrAlSiN does not oxidize when heat treated up to 800ºC and can be applied to high-speed steel tools. (*B4*:A6) The improved properties of these coatings are associated with the fact that they are made of nanocrystals of TiN, $AlN/Cr_xAl_{1-x}N$ with size less than 10 nm in an amorphous matrix of Si_3N_4/SiN_x. On the example of the TiAlSiN coating, a new approach (annealing of the coating immediately after its deposition) is proposed to stabilize the nanostructure, improve the mechanical properties and wear resistance of one of the most commonly used coatings - those based on Ti.

2.3. A new nanostructured CrAlSiN-AlSiN coating with periodically modulated composition was created, which is a nanocomposite consisting of (CrAl)N and (AlSi)N nanocrystals with sizes of 9 and 7 nm embedded in an amorphous matrix of Si_3N_4. (*B4*:A7,A8) This structure provides extremely high hardness (54 GPa), and by modulating the composition, high elastic damage resistance and plastic deformation resistance have been achieved. The coating retains the combination of ultra-high hardness with resistance to elastic stresses and plastic deformations up to 800 °C and this makes it suitable for high-temperature applications.

2.4. Low-temperature technologies have been developed for deposition at <200 ºC of three-component (TiAlN, TiCrN and CrTiN; (*G7*:B9,B11,B12)) and four-component (TiCrAlN and CrTiAlN; (*G7*:B10,B12,B13)) nitrides of transition metals by unbalanced magnetron sputtering. The research in this direction is motivated by practice - the use of low-temperature instrumental steel, on which the deposition of coatings requires low temperatures. The resulting coatings have a stable crystal structure and very good hardness (27 – 32 GPa) for low-temperature technologies. By using a suitable combination of adhesion and gradient transition layer, good adhesion to substrates was also achieved. The CrTiAlN coating has the best combination of mechanical properties and adhesion-cohesion resistance, which is maintained up to 600 °C.

2.5. Lyers from AlN with different structural characteristics were deposited by pulsed laser deposition in vacuum or nitrogen at different nitrogen pressures and parameters of the laser beam, and the substrate temperature was in very wide range (20 – 800 ºC). (*D7*:B14) A relation was found between the deposition conditions and the structure, mechanical and optical properties of the layers, with some layers achieving quite high hardness (up to 30 GPa) and very high resistance to plastic deformation.

The main scientific and applied contributions can be regarded as: (a) creation of new technologies (contributions 1.3, 1.4, 2.1-2.4) and constructions (utility model Г9:C2): (b) proving by new means essential new aspects of already existing scientific problems (1.1, 2.5) and obtaining and proving new and confirmatory facts (in all research); (c) contributions to implementation (utility models *G9*:C1,C3, contribution 1.2) and application of scientific achievements in practice (*G9*:C1).

Notes and recommendations on submitted documents

I don't have any questions or significant critical remarks about the publications with which Assoc.Prof. Dr. L. Kolaklieva participated in the competition, but I will note that I had difficulty due to the difference in the numbering of the articles of the candidate in the Author's contributions, in the Lst of articles and in the dDocument with abstracts of the published papers.

Conclusion

The analysis of the above considerd materials of Assoc. Prof. Dr. L. Kolaklieva showed that her scientific and applied contributions significantly exceed the minimum requirements in the Regulations for ZRAS RB and the related Requirements of BAS and ISSP for occupying the academic position "Professor". In view of the relevance, significance and international recognition of her achievements, I have no doubt that Assoc. Prof. Dr. L. Kolaklieva is very suitable for the academic position of "Professor" according to the announced competition.

I am fully convinced to support the candidacy of Assoc. Prof. Liliana Kolaklieva and recommend to the honorable Scientific Jury to propose to the Scientific Council of ISSP-BAS that Assoc. Prof. Kolaklieva be elected to the academic position of "Professor" in the Central Laboratory of Applied Physics at the Bulgarian Academy of Sciences, professional direction 4.1. "Physical Sciences", scientific specialty "Physics of Condensed Matter".

22.08.2022 Signature:

/Prof.Dr.Sci. D. Nesheva-Slavova/