PONDICHERRY UNIVERSITY

EXECUTIVE SUMMARY OF THE WORK DONE ON THE MAJOR RESAERCH PROJECT

1.	TITLE OF RESEARCH PROJECT (PHYSICS):	**"Non-spherical like Ag nanoparticles decorated on ZnO Nanorods/Ag nanoislands based substrate for the enhanced SERS effect"**
2.	NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR:	**Dr. D. Bharathi Mohan** Assistant professor, Department of Physics, Pondicherry University, Puducherry-605014. d.bharathimohan@gmail.com, bharathi.phy@pondiuni.edu.in 0413-2654786, 9489966376 (M)
3.	NAME AND ADDRESS OF INSTITUTION:	**Pondicherry University** (A Central University) B. R. Ambedkar Administrative Building, R. Venkataraman Nagar, Kalapet Puducherry-605 014, India
4.	UGC APPROVAL LETTER NO. AND DATE:	• *UGC-MRP, F. No. 41-868/2012(SR) Dated July 23, 2012* • *UGC-MRP, F. No. 41-868/2012 (SR) Dated Oct 29 , 2014*
5.	EFFECTIVE DATE OF STARTING OF THE PROJECT:	July 01st 2012
6.	TENURE OF THE PROJECT:	July 01st 2012 to June 30th 2015
7.	TOTAL GRANT ALLOCATED:	Rs. 14,98,300/-
8.	TOTAL GRANT RECEIVED:	Rs. 13,55,999/-
9.	FINAL EXPENDITURE :	Rs. 13,16,352/-
10.	TITLE OF THE PROJECT:	*"Non-spherical like Ag nanoparticles decorated on ZnO Nanorods/Ag nanoislands based substrate for the enhanced SERS effect"*
11.	OBJECTIVE OF THE PROJECT:	• *Fabrication of ultrathin Ag films and annealing them at different temperatures to tune the Surface Plasmon Resonance (SPR) in broad energy region, which is the main optical property supported by Ag nanostructures needed to be tuned for Surface Enhanced Raman Scattering (SERS) application.* • *Fabrication of ZnO nanorod array on tuned ultrathin Ag film and embedding non-spherical like Ag nanoparticles (cubes, rods, triangles etc.) on to the ZnO/Ag composite.* • *Understanding the Ag inter-particle coupling which creates strong confinement of electric field at the vicinity of the composite nanostructure which facilitates the greater SERS enhancement of analytes which could have the capability often to detect single molecule.*
1.	WHETHER OBJECTIVES WERE ACHIEVED (GIVE DETAILS):	YES objectives were achieved. *Ultrathin Ag films were fabricated by using Thermal Evaporation technique and ZnO nanorods were grown on Ag films by hydrothermal method. Ag nanoparticles were decorated on ZnO nanorods array by means of Thermal evaporation. Probe molecules such as Rhodamine-6G, DNA and Glucose were detected on Ag/ZnO/Ag hybrid structure using Raman spectrometer up to the lower concentration level of 10^{-8} M. The crystal structure and optical properties of Ag/ZnO/Ag nanostructures were studied by X-Ray Diffraction (XRD), UV-Vis Spectrophotometer and Photoluminescence Spectrometer. Atomic Force Microscope (AFM) and Scanning*

		Electron Microscope (SEM) techniques have been utilized for the morphological analysis.
2.	ACHIEVEMENTS FROM THE PROJECT:	• *ORAL presentation on "Fabrication of recyclable SERS of Ag/ZnO/Au substrates and thier study on Hemoglobin detection at IUMRS-ICEM 2016, Suntec, Singapore, 4-8, July 2016.* • *BEST POSTER AWARD received on paper entitled "Label Free Detection of DNA on Au/ZnO/Ag Hybrid Structure Based SERS Substrate" at 2nd International Conference on Emerging Technologies: Micro to Nano, Manipal University, Jaipur, 24-25 Oct. 2015.* • *BEST POSTER AWARD received on paper entitled "Label Free Detection of DNA on Au/ZnO/Ag Hybrid Structure Based SERS Substrate, at 2nd International Conference on Emerging Technologies: Micro to Nano, Manipal University, Jaipur, 24-25 Oct. 2015.*
3.	SUMMARY OF THE FINDINGS (IN 500 WORDS):	***1. Structural, morphological and optical properties of Ag–AgO thin films with the effect of increasing film thickness and annealing temperature.*** *Ag films of thickness ranging from 5 to 60 nm were deposited by thermal evaporation technique followed by air annealing process with temperature varying from 50 to 250 °C. Morphological properties such as particle size, shape, surface roughness and number particles density were studied by atomic force microscope (AFM). The structural transition from quasi-amorphous to nanocrystalline to crystalline upon increasing film thickness and annealing temperature were studied. Ag films with smallest particle size and surface roughness were achieved up to film thickness of 7 nm. The possibility of surface oxidation of Ag on both as deposited and annealed films was studied through Raman mapping by using confocal Raman spectroscopy. Ag film was X-ray amorphous even after annealing process up to the film thickness of 7 nm and above which the crystallinity reached maximum at 250 °C. The surface plasmon resonance (SPR) with a symmetric line shape due to dipole–dipole interactions was found to be very strong for film thickness of 5 nm at 100 °C, attributed to the formation of smaller Ag NPs size of ~22 nm with least size distribution and higher particles number density of ~1625 μm^{-2} in a self-organized fashion. With an increase of film thickness and annealing temperature, an asymmetric broad absorption arose due to increase in damping of collective electron oscillation on bulky NPs. Theoretical absorption spectra were simulated using extended Maxwell garnet method showing a decent agreement with experimental data. The real and imaginary parts of dielectric constants were determined and plotted for different film thick nesses of as deposited Ag films. Even though the film is oxidized at the surface level, it still can be used for plasmonic sensor applications however the film thickness should be approximately 7 nm for the enhanced result.* ***2. Fabrication of partially oxidized ultra-thin nanocrystalline silver films: effect of Surface Plasmon Resonance on Fluorescence Quenching and Surface Enhanced Raman Scattering*** *Plasmonically active nanocrystalline silver (Ag) films of mass thickness ~1 nm was fabricated using thermal evaporation followed by air annealing process temperature varying from 50 °C to 250 °C. The effect of air annealing on surface morphology and optical absorbance of quasi-amorphous and nanocrystalline Ag films was studied. The possibility of formation of silver oxide (AgO) at the surface level of Ag nanostructures due to air annealing was studied using confocal Raman Spectrometer. Surface Enhanced Raman Scattering (SERS) enhancement and its reverse mechanism known as fluorescence quenching correspond to Rhodamine-6G (Rh6G) molecules were studied on Ag films through altering crystallinity, interparticles distance, size distribution and number density of Ag nanopartices (NPs).*

		3. ZnO/Ag Composite Nanorod Arrays for Surface-Plasmon-Enhanced Emission Study *The surface plasmon resonance enhanced emission through coupling of surface plasmons and exciton band energies was studied in hybrid ZnO/Ag nanostructure. The catalytic growth of ZnO nanorods was controlled in seed mediated growth by altering size distribution of Ag nanoislands. X-ray diffraction showed a predominant (002) crystal plane confirming the preferential growth of ZnO nanorods on as-deposited Ag. Increase of surface roughness in Ag film by post deposition annealing process enhanced the light emission due to momentum matching between surface plasmons and excitons as well as a red shift of 32 meV occured due to multi phonon and phonon-exciton interaction.* ***4. Study of NBE emission enhancement of ZnO nanorods by changing the surface property of ultra-thin Ag interlayer.*** *ZnO nanorods (NRs) exhibiting enhanced ultra-violet near band edge (UV-NBE) emission without a broad visible deep level (DL) emission has been investigated on catalytically grown ZnO/Ag hybrid nanostructure. The hybrid structure is fabricated in two steps, (1) Thermal evaporation of ultra-thin catalytic layer of Ag with mass thickness ~1 nm on glass substrate followed by annealing process from 50 to 250 °C and (2) vertical growth of ZnO NRs by hydrothermal reaction process on all Ag films. The surface properties of Ag layer such as particle size, inter-particle distance, particles number density, surface roughness and surface coverage area were altered through annealing process. Annealing at 100 °C modifies Ag from quasi-amorphous to nanocrystalline leading to high density growth and high aspect ratio of ZnO NRs where as a random and less density growth was realized at 250 °C due to increase of both particle size and inter-particles distance in Ag layer. X-ray diffraction reveals a predominant growth of (002) plane at 100 °C confirming the formation of wurtzite phase of ZnO NRs with highest texture coefficient of 2.35. Raman spectra verify the chemical structure of ZnO with very good crystallinity. Absorption spectra demonstrates the overlapping of surface plasmon resonance (SPR) and exciton bands up to 200 °C while the excitonic absorption band is resolved at above 200 °C because of the red shift in SPR due to change in surface properties of Ag layer. At 250 °C, a broad optical absorption spectra from 300 to 800 nm attributed to the dominant properties of SPR and exciton. Besides acting as a catalyst, Ag interlayer enhances the NBE emission at above 200 °C through electrons transfer from Ag to ZnO which is quite possible because of the direct contact between them, explained by giving energy band diagram. The morphology is such that there is an increase in passage for light interaction due to less density and random growth of ZnO NRs leading to increase light scattering over absorption of Ag interlayer.* ***5. The study of surface plasmon enhanced emission of ZnO nanorods on plasmonic Ag nanorods array*** *Ag islands and nanorods (NRs) array were fabricated through glancing angle deposition (GLAD) method at an angle of 85 °C by using thermal evaporation technique. Subsequently, ZnO NRs were grown along the side of Ag NRs arrays through hydrothermal reaction. The crystal structure of ZnO/Ag hybrid structure was studied by Glancing Incident X-Ray Diffraction (GIXRD). The surface morphologies of Ag NRs arrays and ZnO NRs were studied through AFM and SEM techniques. The presence of different vibrational energy modes of ZnO structure was studied through confocal Raman spectrometer. The surface plasmon enhanced fluorescence of ZnO NRs was probed through fluorescence spectrophotometer. The presence of strong UV emission and the absence of intense visible emission of ZnO NRs are due to the existence of fewer crystal*

		defects which could make the hybrid structure precious for UV LEDs. ***6. SERS detection of D-Glucose on ultra-thin Ag-Cu films through tuning of Surface Plasmon Resonance*** *Ultra-thin Ag-Cu films of 7 nm thicknesses are prepared by thermal evaporation technique followed by post deposition vacuum annealing process at 200 ℃ 1 hour. Different weight percentages of Ag and Cu such as 95% and 5% respectively were grinded using an agate mortar, pestle and used as source for thin film preparation. The AFM imaging of corresponding films shows the surface morphology, particle size and RMS surface roughness of ultra-thin Ag-Cu films. The vacuum annealing process directs the formation of nano-crystalline phase from quasi amorphous phase of ultrathin Ag-Cu films. Optical absorption studies shows the tuning of surface Plasmonic resonance of Ag-Cu thin films in the mid-range of SPR of pure Ag (457 nm) and SPR of pure Cu (570 nm). Detection of D-Glucose molecules of very low concentration 10^{-3} M is achieved on ultra-thin Ag-Cu films. The SERS enhancement of D-Glucose is discussed based on tuning of SPR of Ag-Cu films through alternating crystallinity, particle size, inter-particle distance and number density of Ag-Cu nanoparticles.* ***7. SERS Enhancement of Glucose Molecules on Layered Hybrid Ag/ZnO/Ag Nanostructure*** *Ag/ZnO/Ag hybrid structure was fabricated for the detection of D-glucose molecules. Photoluminescence spectra revealed the quenching due to Ag decreases the passivation of emitted light from ZnO. The reasons for enhancement, (i) the strong local electric field at nanometer gap between Ag nanoparticles, (ii) the hydrophobic surface of ZnOnanorods and (iii) the amplified electric field due to charge transfer from Ag to ZnO.* ***8. Ag Nanoparticles Decorated on Zno Nanrods Array Based SERS Substrate for Label Free Detection of DNA*** *Label free detection of DNA of Escherichia Coli bacteria was achieved on Ag nanoparticles decorated on ZnO nanorods array based Surface Enhanced Raman Scattering (SERS) substrate. Scanning electron microscope (SEM) cross sectional image corroborates the decoration of Ag nanoparticles (NPs) on ZnO nanorods (NRs) array. X-ray diffraction (XRD) pattern showed the formation of composite structure of Ag/ZnO/Ag hybrid structure. Confocal Raman spectroscopy study confirms the detection of DNA molecules even at lower concentration level of 10^{-6} M.*
4.	CONTRIBUTION TO THE SOCIETY (GIVE DETAILS):	*The research on this project has been resulted in award of PhD degree to one scholar and 3 have been enrolled and thus contributed the society in imparting education and skill in surface enhanced Raman spectroscopy and enabling them to address advanced scientific challenges. The society is facing challenges towards early diagnosis of cancer cells and food borne deceases. If the sensitivity of the SERS increases, it will be highly stress-free in detecting new deceases. The fabrication is successfully carried out in my lab and tested with hemoglobin and DNA molecules. This kind of ultra sensitive SERS substrates could be very useful for development of SERS based biosensors in near future.*
5.	WHETHER ANY PH.D. ENROLLED/ PRODUCED OUT OF THE PROJECT:	*One PhD degree awarded and one enrolled.*

6.	NUMBER OF PUBLICATIONS OUT OF THE PROJECT (PLEASE ATTACH):	***Publications in International Journals:*** *1.A.K. Pal and D.B. Mohan, Fabrication of partially oxidized ultra-thin nanocrystalline silver films: effect of surface plasmon resonance on fluorescence quenching and surface enhanced Raman scattering, Materials research Express 1, 025014 (2014) .* *2.A.K. Pal and D.B. Mohan, Study of NBE emission enhancement with an absence of DL emission from ZnO nanorods through controlled growth on ultra-thin Ag films, Applied Surface Science 333, 244-253 (2015).* *3.A.K. Pal and D.B. Mohan, Structural, morphological and optical properties of Ag–AgO thin films with the effect of increasing film thickness and annealing temperature, Optical Materials 48, 121-132 (2015).* ***Conference Proceedings:*** *1. A.K. Pal and D.B. Mohan, ZnO/Ag Composite Nanorod Arrays for Surface-Plasmon-Enhanced Emission study, AIP Conference Proceeding 1591, 982-984 (2014).* *2. A.K. Pal and D.B. Mohan, Study of NBE emission enhancement of ZnO nanorods by changing the surface property of ultrathin Ag interlayer, IEEE Xplore, DOI.10.1109/ICEmElec.2014.7151155 (2015).* *3. A.K. Pal and D. Bharathi Mohan, The study of surface plamon enhanced emission of ZnO nanorods on plasmonic on plasmonic Ag nanorods array, Accepted for publication in Procedia Materials Science, December 2014.* ***Contributions in National/ International Conferences:*** *1.A. K. Pal, M. T. Abhaya and D. B. Mohan, SERS detection of D-Glucose on ultra-thin Ag-Cu films through tuning of Surface Plasmon Resonance, presented as ORAL presentation at Recent Advances in Nano Science and Technology (RAINSAT), Satyabama University, Tamil Nadu, 8-10, July 2015.* *2.A. K. Pal and D. B. Mohan, Study of NBE emission enhancement of ZnO nanorods by changing the surface properties of ultrathin Ag interlayer, presented as POSTER presentation at 2nd IEEEInternational Conference on Emerging Electronics, (ICEE), IISc, Bangalore, 3-6, Dec. 2014.* *3.A.K. Pal and D.B. Mohan, Study of surface Plasmon enhanced emission of ZnO nanorods on plasmonic Ag nanorodarrays, presented as ORAL presentation at International Conference on Nano Science & Engineering Applications (ICONSEA), CNST, JNTU, Hyderabad, 26-28, June 2014.* *4.Anil Kumar Pal and D. Bharathi Mohan, ZnO/Ag Composite Nanorod Arrays for Surface-Plasmon-Enhanced Emission Study, presented as POSTER presentation at DAE-SSPS, Thappar University, Patiala, 17-21, Dec. 2013.* *5.Anil Kumar Pal and D. Bharathi Mohan, Surface Plasmon and Exciton Interaction in ZnO/Ag Nanocomposite Structure, presented as ORAL presentation at IUMRS-ICA, IISc, Bangalore, 16-20, Dec. 2013.* *6.Anil Kumar Pal and D. Bharathi Mohan, SPR Tuning in Ultra-Thin Ag-AgO Films for SERS Application, presented as POSTER presentation at ICANN, IIT Guwahati, 8-10, Dec. 2013.* *7.Anil Kumar Pal and D. Bharathi Mohan, Fabrication and Characterization Studies of Silver Nanocubes based Surface Enhanced Raman Spectroscopy Substrate, presented as POSTER presentation at National Conference on Advanced Materials & Applications- NCAMA2013, NIT Trichy, 4-5, Apr. 2013.*

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PRINCIPAL INVESTIGATOR
Dr. D. BHARATHI MOHAN
Assistant Professor
Department of Physics
Pondicherry University
Pondicherry - 605 014, India

ASSESSMENT/EVALUATION REPORT OF MAJOR RESEARCH PROJECT

Sponsored by

University Grant Commission

Bahadur Shah Zafar Marg

A. DETAILS OF PROJECT

Title of the project	: **"Non-spherical like Ag nanoparticles decorated on ZnO Nanorods/Ag nanoislands based substrate for the enhanced SERS effect"**
Total duration of project	: **3 years** ***(July 2012 to June 2015)***
Project status	: **Completed**
Subject	: **Physics**
File Number (Ref. No)	: ***F.No.41-868/2012 (SR) dated 23 July 2012***
Grants Approved	: **Rs.14,98,300/-** ***(Fourteen Lakhs Ninety Eight Thousand Three Hundred Only)***

B.EVALUATION REPORT OF EXPERT MEMBER

1.	Name of Principle Investigator	: ***Dr. D. Bharathi Mohan***
2.	Designation	: Assistant professor
3.	Address of Principle Investigator	: Department of physics Pondicherry university Pondicherry-605014
4.	Whether work is focused on the title of the sanctioned project	: YES
5.	Whether original work is done	: YES
6.	Whether significant contribution made by the Principle Investigator	: YES
7.	Whether proposed work have relevance to the society/scientific community	: YES
8.	What type of contribution found in the final report: theoretical/practical. If there are theoretical contributions given by The Principle Investigator whether real applications are given	: Experimental and Theoretical

9.	Whether theoretical contributions and their results and finds are published	: YES
10.	Whether results and findings are significant	: YES
11.	Whether the significant publications made by principle investigator in peer reviewed journals	: YES
12.	The number of publications made by principle investigator in standard reputed journals	: Three (03)
13.	Whether contributions made by principle investigator is sufficient	: YES
14.	The finding and results of the sanction major research projects are justifiable	: YES
15.	Whether completed project work meet the proposed objectives	: YES
16.	Give your brief comments on the overall work of the project	: The objectives of the project were sucessfully achieved. * The quality of work is highly appreciable.
17.	Any specific comments	: PI Sucessfully fabricated SERS substrates from Ag based ZnO.
18.	Indicate your overall assessment of the project ~~poor/good~~/excellent	: Highly satisfactory and recommended for final submission to UGC

Date: 08/03/2019

Place: MIT; Chromejet

R. Murugaraj
08/03/19
Signature

Dr. R. MURUGARAJ
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Name and Address of Expert

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Registrar
Pondicherry University

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B.EVALUATION REPORT OF EXPERT MEMBER

1.	Name of Principle Investigator	: ***Dr. D. Bharathi Mohan***
2.	Designation	: Assistant professor
3.	Address of Principle Investigator	: Department of physics, Pondicherry university Pondicherry-605014
4.	Whether work is focused on the title of the sanctioned project	: YES
5.	Whether original work is done	: YES
6.	Whether significant contribution made by the Principle Investigator	: YES
7.	Whether proposed work have relevance to the society/scientific community	: YES
8.	What type of contribution found in the final report: theoretical / practical. If there are theoretical contributions given by The Principle Investigator whether real applications are given	: Experimental and Theoretical
9.	Whether theoretical contributions and their results and finds are published	: YES
10.	Whether results and findings are significant	: YES
11.	Whether the significant publications made by	: YES

	principle investigator in peer reviewed journals	
12.	The number of publications made by principle investigator in standard reputed journals	: Three (03)
13.	Whether contributions made by principle investigator is sufficient	: YES
14.	The finding and results of the sanction major research projects are justifiable	: YES
15.	Whether completed project work meet the proposed objectives	: YES
16.	Give your brief comments on the overall work of the project	: Ag based 2nD-SERS substrates were fabricated with very good enhancement factor.
17.	Any specific comments	: Overall the objectives of the project was achieved successfully which is reflected in Papers.
18.	Indicate your overall assessment of the project poor/good/excellent ✓	: Excellent.

Date: 15.03.2019

Place: KARAIKAL

Signature

Name and Address of Expert

Dr. Ajay Kumar Mishra
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NIT Puducherry, Karaikal

(Principle Investigator)

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