# Research Interests:

- Ultra clean surfaces using MBE, RHEED, VTSTM (in-situ): Si-Ge, Au-Si, Ag-Si
- MBE growth on high index substrate orientations (Ag, Ge on Si(5 5 12), Si ( 5 5 3).
- Computational methods for understanding various structural phase transformations
- Various Imaging methods: SEM, HRTEM, FIB and X-ray Microprobes
- Structure – Property establishment of thin films and in nanostructures
- Surface and Interfaces: HRTEM, SEM, STEM, RBS, Channeling, Synchrotron X-ray methods
- Real time in-situ electron microscopy of metal-semiconductor interfaces
- Nanopatternning on surfaces: Nanotechnology (FIB, E-beam lithography)
- Nucleation and growth studies related to self-organized growth
- Metal – Semiconductor Surfaces and Interfaces
- Fabrication and modification of nanostructures using Ion Beams (keV to MeV)

## 2. Consolidated Report on Academic Activities in last five (5) years ...

In last about five years, our focus was to concentrate on in-situ growth and structural characterization of nanostructures under MBE (UHV) conditions and other physical vapor deposition and chemical vapor deposition methods. Modification of the structures under equilibrium conditions (heating/annealing) and non-equilibrium conditions) and study these modifications using *insitu* TEM and insitu XRD (using synchrotron radiation methods) had been on focused themes. We have used in-house facilities such as HRTEM along with low/high temperature facilities, *insitu* XRD (at KEK Photon factory, Indian Beam line and HASYLAB at DESY beam time at Hamburg) and MeV ion scattering measurements at IOPB. Under $11^{th}$ Plan Project, as principal investigator, I have set up a sophisticated dual beam (cross-beam) facility with Focused Ion Beam and Field Emission Gun based Scanning Electron Microscopy and using this facility two Ph.D. thesis works (several publications) have been obtained. Also, as a part of $11^{th}$ Plan project, I have set up insitu-TEM facility by incorporating a high temperature stage (RT – 1000 C) and a low temperature stage ($LN_2$ to 100°C) along with a fast CCD for real time high resolution TEM imaging. During my one year sabbatical stay at University of Bremen, Germany, I worked with advanced TEM methods (using aberration correction TEM) for understanding the quantum structures.

### (i) Ion Beam Induced Surface and Interface Modification of Nanostructured thin films on silicon substrate

Thin films of gold and silver of various thicknesses have been deposited using thermal evaporation method. At lower thickness, a complete wetting of silicon substrate is not possible and this results in formation of nanostructured thin films. The effects of mass, energy, fluence, flux and impact angle of impinging energetic ions and density and temperature of the substrate have been studied. The focus in this part of the work was on the investigation of the various aspects of ion beam interaction with nanostructured films a function of incident beam flux and fluence. The ion induced effects that are studied in detail are: (a) Sputtering yield (b) Sputtered particle size distribution (b) Crater formation (c) Ion beam mixing leading to nano-scale alloy formation at surface and interfaces (d) Enhanced diffusion leading to dramatic mass transport (e) Segregation (f) embedding and (g) strain at interfaces. For all ion irradiation effects studied in this thesis work, only 1.5 MeV $Au^{2+}$ ions are used.

**Selected publications:**

1. Synchrotron X-ray induced damage in polymer (PS) thin films,
   Umananda M. Bhatta, J. Ghatak, M. Mukhopadhyay, Jin Wang, Suresh Narayan and **P. V. Satyam***
   Nuclr. Instrum. Meth. In Phys. Res. B267 (2009)1807

2. Strain evolution in Si substrate due to implantation of MeV ions observed by extremely asymmetric x-ray diffraction,
   T. Emoto, J. Ghatak, **P. V. Satyam** and K. Akimoto,
   J. Appl. Phys. 106 (2009) 043516

3. MeV ion induced modifications at Co/Si interface
   J. Ghatak, D. Kabiraj, and **P. V. Satyam**
   Appl./ Surf. Science 256 (2009) 572

4. Evolution of surface morphology of NiO thin films under swift heavy ion irradiation
   P. Mallick, Chandana Rath, S. Majumde, R. Biswal, D.C. Agarwal, Shikha Varma, D.K. Avasthi, **P.V. Satyam**, and N.C. Mishra,
   Appl. Surf. Sci. 256 (2009)521

5. Surface roughness and power spectral density study of SHI irradiated ultra-thin gold films , P. Dash, P. Mallick, H. Rath, A. Tripathi, Jai Prakash, D.K. Avasthi, S. Mazumder, S. Varma, **P.V. Satyam** and N.C. Mishra
   Appl. Surf. Sci. 256 (2009)558

6. Effect of thermal and athermal processing on the formation of buried SiC layers,
   Y. S. Katharria, Sandeep Kumar, D. Kanjilal, Devki Chauhan, J. Ghatak, U. Bhatta, and **P. V. Satyam**
   J. Appl. Phys. 105, 014301 (2009)

7. Ph.D. Thesis of Dr. J. Ghatak

**(ii) Temperature dependent TEM studies of gold nanostructures on silicon surfaces:**

This involves various aspects related to thermal behavior of gold nanostructures in two different systems: (i) on single crystalline silicon substrates of various orientations. The emphasis in this work is on the use of transmission electron microscopy (TEM) for carrying out temperature dependent structural variations in gold nanostructures in inorganic (silicon). Through this program, we established ourselves as one of leading groups in In-situ and Real time TEM studies" in the world (and among very few groups in India). In this work gold thin films of different thickness were deposited on Si(110) substrates both UHV-MBE and thermal evaporation methods. In the case of MBE samples (1.0, 2.0, 5.0 and 11.7 nm) (*this represents for the case where no native oxide is present at Au-Si interface*), upon in-situ annealing in TEM hot-specimen-stage, as deposited irregular nanostructures underwent a shape transformation to form well aligned gold silicide nanorods all aligned along [1-10] direction at higher temperatures (≈ 700˚C) (for 1.0 and 2.0 nm case). The selected area diffraction taken at room temperature confirmed the formation of $Au_5Si_2$. The alignment of gold silicide structures has been explained on the basis of lattice matching between the substrate silicon and silicide structures. In the case of higher thickness (5.0 and 11.7 nm), formation of much bigger structures aligned in the same directions were found. In presence of a native oxide layer gold coverage was much lesser as compared to MBE sample. Formation of aligned nanostructures were found for all three thickness namely: 2.0, 5.0 and 11.7 nm. Aspect ratios were much higher (as high as 15) in the case of 2.0 nm $Au/SiO_2/Si(110)$ system [3]. Similar formation of aligned nanostructures along [1-10] were found for higher thickness as well. The growth of these gold silicide nanostructures along [1-10] direction has been explained based on selective thermal decomposition of native oxide layer followed by liquid-solid growth mechanism. As a comparative study, similar measurements were done on 2.0 nm Au/Si(111) system in presence of a native oxide layer. Formation of facetted structures were observed following the 3-fold symmetry of the substrate.

**Selected Publications:**

1. Structural phase transitions in au thin films on Si(110): An in-situ temperature dependent transmission electron microscopy study
   Umananda M Bhatta, J. K. Dash, A. Rath, and P. V. Satyam
   Appl. Surf. Sci. 256 (2009) 571

2. Oxide mediated liquid - solid growth of high aspect ratio – aligned gold silicide microrods on Si (110) substrates,
   Umananda M Bhatta, A. Rath, J. K. Dash, and P. V. Satyam
   Nanotechnology 20 (2009) 465601

3. Formation of aligned nano-silicide structures in MBE grown Au/Si(110) system: A real time temperature dependent TEM study,

UmanandaM Bhatta, J K Dash, Anupam Roy, A Rath and P V Satyam
J. Phys.: Condens. Matter 21 (2009) 205403

4. Ph.D. thesis of Dr. Umananda M Bhatta (2010)

### *(iii)* Dynamic and static TEM studies on the formation of Au-Si and Au-Ge Nanostructures

This work involves an extensive study on the formation of Au-Si and Au-Ge nanostructures under different thermal annealing conditions achieved by varying parameters such as, pressure, film thickness, substrate, presence or absence of native oxide on the substrate, temperature etc. In this study, my group made interest results on the growth of highly oriented Au nano/micro structures with simple annealing procedure, such as annealing thin films of Au on Silicon substrate at high temperature but in ambient conditions such that growth of oxide layer interface is dominant enough to forbid the inter-diffusion of Au in Si. In this work, the use of bottom-up nano particle self-assembly techniques to synthesize well tuned Au, Ge, Au-Si, and Au-Ge alloyed nano structures has been studied. Our results suggest that, by varying the parameters such as temperature and interface conditions, we can form Au-Si and Au-Ge nano-structures of various shapes and sizes which can be used as catalysts for growing various crystalline nanowires. The range of the study covers thin film thickness from as low as 2.0 nm to 50 nm of gold and germanium. The emphasis in was on the use of transmission electron microscopy (TEM) for carrying out in-situ temperature dependent structural variations in Au and Ge nanostructures and their respective alloys on silicon and germanium substrate.

**Selected Publications:**

1. Temperature dependent electron microscopy study of Au thin films on Si(100) with and without native oxide layer as barrier at the interface
   A. Rath, J. K. Dash, R. R. Juluri, A. Rosenauer and P.V. Satyam
   J. Physics D: Applied Physics, 44 115301 (2011)

2. Nano scale phase separation in Au-Ge system on ultra clean Si(100) surfaces.
   A. Rath, J. K. Dash, R. R. Juluri, Marco Schowalter, Knut Mueller, A. Rosenauer and P.V. Satyam
   J. Appl. Phys. 111, 104319 (2012) ;arXiv:1202.0614v1

3. Growth of Oriented Au Nanostructures: Role of Oxide at the Interface
   A. Rath, J. K. Dash, R. R. Juluri, A. Rosenauer, Marcos Schoewalter and P.V. Satyam
   J. Appl. Phys. 111, 064322 (2012);arXiv:1203.0819v1

4. Dynamic and Static Transmission Electron Microscopy Studies on Structural Evaluation of Au nano islands on Si (100) Surface.
   A. Rath, R. R. Juluri and P.V. Satyam ; arxiv:1204.4618

5. Structural modification in Au/Si (100) system: Role of surface oxide and vacuum level
   A. Rath, J. K. dash, R. R. Juluri and P. V. Satyam. ; arXiv:1204.5370

6. Study of initial stages of growth of Au-assisted epitaxial Ge nanowires on clean Ge(100) Surface
   A. Rath, J. K. dash, R. R. Juluri, A. Ghosh and P. V. Satyam.
   Crystal Engineering Communications (2013) (Accepted For Publications)

7. Ph.D. thesis of Dr. A. K. Rath (2012)

### *iv)* Growth of graded $Si_{1-x}Ge_x$ nano/micro structures on high index silicon surfaces

One of the major problem issues that need to be understood is to understand the structural evaluation of low-dimensional nanostructures growth on patterned surfaces. This study helps in combining the bottom-up process to the top-down process. We have a project undergoing ($11^{th}$ Plan and now $12^{th}$ Plan proposal funded by DAE) on the nanostructure growth on the patterned surfaces. One can use different methods to create patterned surfaces: Patterning by nano-electron or ion beam (e-beam or focused ion beam lithography) or using the natural self-assembled steps on high vicinal or/and high index plane surfaces. Our group has found very interesting results on the growth of aligned trapezoid structures to core-shell structures of Si-Ge on ultra clean Si(5512) surfaces. We have used Molecular Beam Epitaxy (MBE), *insitu* Reflection High Energy Electron Diffraction (RHEED), in-situ TEM, FEGSEM and FIB for growth and characterization of interesting Ge-Si nanostructures. We have used high index Silicon surfaces like Si(5512), Si(557), Si(553) etc. for the growth of controlled aligned nanostructures. Si( 5 5 12) is a stable vicinal surface of large unit cell. Since last year my main focus has been on Growth of Ge on Si(5 5 12) with different coverage and substrate temperatures. We have deposited 1.0 monolayer (ML), 2.0 ML, 3.0 ML, 5.0 ML and 10.0 ML Ge on Si(5 5 12) in MBE at substrate temp. $600^{o}C$ followed by a post anneal of 15 minutes at the same temperature. In all the cases the samples were in direct heating mode with current passing along the step edges to avoid step bunching. All the samples have been characterized with FEG-SEM and HRTEM and FIB-SEM mode for 3D reconstruction in Ge-Si nanostructures. We found aligned trapezoid structures of different dimensions on all the samples depending upon the coverage. In the 10 ML Ge/Si(5 5 12) system Ge-Si Core shell like structures have been found in TEM micrographs and the compositional analysis of core & shell is under process. Presently the statistical distribution of aligned nanostructures with different coverage's & the TEM analysis is being done. Scanning transmission electron microscopy (STEM) measurements suggested the mixing of Ge and Si. Synchrotron XRD has confirmed the formation of graded SiGe alloys.

We have carried out the theoretical modeling to complement the shape evolution observed in the experiments. The theoretical model includes 2D kinematic Monte Carlo (kMC) simulation used to explain the phenomena of shape transition observed in the experiments by introduce anisotropy through binding energies of different types of bonds and the dependence of surface barrier on the direction of hopping.

**Selected Publications:**

1. Shape transformation of Si1-x Gex structures on ultra clean Si (5 5 7) and Si (5 5 12) surfaces
   J. K. Dash, A. Rath, R. R. Juluri,P Santhana Raman, K. Muller,M. Schowalter, R.Imlau, A. Rosenauer and P.V. Satyam
   Journal of Physics: Conference Series 326, 012021(2011)

2. DC heating induced shape transformation of Ge structures on ultra clean Si (5 5 12) surfaces
   J. K. Dash, A. Rath, R. R. Juluri,P Santhana Raman, K. Muller, A. Rosenauer and P.V. Satyam
   J. Phys.: Condens. Matter 23,135002 (2011).

3. Shape evolution of MBE grown Si_{1-x} Ge_{x} structures on high index Si(5 5 12) surfaces: A temperature dependent study.
   J. K. dash, A. Rath, R. R. Juluri and P. V. Satyam.
   J. Phys. D: Appl. Phys. 45 (2012) 455303.

4. Universality in Shape Evolution of Si_ {1-x} Ge _ {x} Structures on High Index Silicon Surfaces
   J. K. dash, T Bagarti, A. Rath, R. R. Juluri and P. V. Satyam.
   Europhys. Lett. 99, 66004 (2012)

5. Ph.D. thesis of Dr. J. K. Dash (2012)

**(v) Coherently Embedded Ag Nanostructures in Si: 3D Imaging and their application to SERS**

Surface enhanced Raman spectroscopy (SERS) has been established as a powerful tool to detect very low-concentration bio-molecules. One of the challenging problems is to have reliable and robust SERS substrate. Here, we report on a simple method to grow coherently embedded (endotaxial) silver nanostructures in silicon substrates, analyze their three-dimensional shape by scanning transmission electron microscopy tomography and demonstrate their use as a highly reproducible and stable substrate for SERS measurements. Bi-layers consisting of Ag and GeOx thin films were grown on native oxide covered silicon substrate using a physical vapor deposition method followed by annealing at 800°C under ambient conditions resulted in the formation of endotaxial Ag nanostructures of specific shape depending upon the substrate orientation. These structures are utilized for detection of Crystal Violet molecules of $5x10^{-10}$ M concentrations. These are expected to be one of the highly robust, reusable and novel substrates for single molecule detection.

We report a novel method of growth for endotaxial silver nanostructures on Si (100), Si (110), Si (111) substrates using a chemical vapor deposition (CVD) and physical vapor deposition (PVD) methods. Our procedure involves a low-temperature thermal etching of native oxide on silicon substrate using $GeO_x$ layer as an etchant to grow substrate symmetry driven endotaxial nano structures in the atmospheric pressure CVD system. A control over the shapes and sizes of the Ag nanostructures has been obtained using the parameters such as substrate orientation and time of deposition during the growth. High resolution electron microscopy has been used to elucidate the growth mechanism of these structures.

**Selected publications:**

1. Substrate Symmetry Driven Endotaxial Silver Nanostructures by Chemical Vapor Deposition
   R. R. Juluri , A. Rath , A. Ghosh , and P. V. Satyam
   J. Phys. Chem. C, 2013, 117 (2013)13247

2. Coherently Embedded Ag Nanostructures in Si: 3D Imaging and their application to SERS
   R R Juluri , A Rath , Mr. A Ghosh , Mr. A Bhukta , Dr. Sathyavathi R , Prof. Narayana rao D , Dr. Knut Mueller , Mr. Kristian Frank , Dr. Marco Schowalter , Dr. Tim Grieb , Mr. Florian Krause , Andreas Rosenauer
   Scientific Reports (Nature) (2014) (in Press)

3. Study of Buried Interfaces during the Silver Endotaxy in Silicon: Role of Ambience during Annealing Process
   R. R. Juluri, A. Ghosh, A. Bhukta and P. V. Satyam
   J. Appl. Phys (2013) (under review)

4. Uniformity of epitaxial nanostructures of $CoSi_2$ via defect control of the Si (111) surface
   J.C. Mahato, Debolina Das, Anupam Roy, R. Batabyal, R.R. Juluri, P.V. Satyam , B.N. Dev, Thin Solid Films 534 (2013) 296

5. Ph.D. Thesis - R. R. Juluri (2014)

**(VI) Oxide Nanowires: Growth, Structure, Characterization and their uses ...**

We report a single step growth process of faceted Au nanoparticles (NPs) on highly c-axis oriented ZnO nanowires (NWs) and report that, a system with a lower anti-reflection coefficient also showed higher surface enhanced Raman spectroscopy (SERS) enhanced factors. Well dispersed Au NPs are grown on silicon substrate using thin film-in-air-annealing method (using 1 nm and 5 nm thick Au films on silicon and subsequent annealing in air at 800 °C) wherein enhanced oxide growth at the Au-Si interface was used to inhibit inter-diffusion to avoid Au-Si alloy formation (Au/SiOx/Si). These substrates are used to grow aligned ZnO nanowires (NWs) using a high temperature (≈900 °C) chemical vapor deposition (CVD) method. Depending on the size and areal density of initial catalytic Au NPs, the resultant photoluminescence (PL), reflectance characteristics, and effectiveness as SERS substrates of the faceted Au NP capped ZnO NWs coatings are systematically studied. The highly oriented and faceted Au NPs on ZnO NWs have been used as free standing surface enhanced Raman spectroscopy (SERS) substrates to detect sub-micro molar crystal violet (CV) molecules with an analytical enhancement factor (AEF) of ≥ 104 and with high repeatability. The substrate with high-density Au-ZnO heterostructures (5 nm Au case) found to have larger AEF, very low reflectance (≈ 0.75%) and more green emission.

Publications:

1. Study of Faceted Au nanoparticle capped ZnO nanowires: Antireflection, Surface Enhanced Raman Spectroscopy and Photoluminescence aspects
A. Ghosh, R.R.Juluri, P.Guha, R.Sathyavathi, Ajit Dash, B.K. Jena and P. V. Satyam
Journal of Physics D: Applied Physics (2014) (Under Review)

Thesis work: Arnab Gosh

**VII High Index Surfaces: Tailoring the Self-assembly with bi-metallic alloys:**

We study the role of Ag or Au on the formation of well ordered high-aspect ratio of Au or Ag nanowires on Si (5 5 12), Si(2 2 7) and Si (5 5 3) surfaces, under various conditions.

Thesis of Mr. Anjan Bhukta