NFAPT Newsletter

NFAPT
NATIONAL FACILITY FOR ATOM PROBE TOMOGRAPHY

Second Newsletter of National Facility for Atom Probe Tomography

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National Facility for Atom Probe Tomography (NFAPT)

The main objective of National Facility for Atom Probe Tomography (NFAPT) is to improve the quality of nano materials research and development in the country with the help of atomic level characterization in 3-dimensions using Local Electrode Atom Probe (LEAP). NFAPT houses the state-of-the-art LEAP (Cameca, LEAP 5000 XR) along with Dual Beam Scanning Electron Microscope (FEI, Helios G4 UX) for LEAP sample preparation and a Transmission Electron Microscope (FEI, Tecnai T12).

Design and development of materials with tailored properties for specific applications requires understanding of elemental distribution at the atomic scale. Atom Probe Tomography (APT) is the only experimental tool to provide three-dimensional elemental distribution in materials at the atomic scale. LEAP has an extremely fast acquisition rate of atoms from the sample for good statistical analysis and can work for a wide range of materials. There are about 100 LEAP facilities all over the world, there was no LEAP in India until 2017. Over the years, India was awaiting its own LEAP and Indian researchers had to depend on collaborators from abroad after sending samples for research at the frontiers of materials science. Realizing the need to set up a LEAP facility that can cater to the advanced materials characterization needs of Indian materials research community, NFAPT has been set up at IIT Madras. This is the first remotely operable near-atomic scale material characterization facility of the country. The NFAPT was inaugurated by Prof. Ashutosh Sharma, Secretary, DST and Prof. Bhaskar Ramamurthi, Director, IIT Madras, in presence of Dr. Thomas F. Kelly, Inventor of LEAP, on July 16, 2018.

This is also possibly for the first time when eight partner institutions in the country have contributed financially to set up such a national facility at a cost of nearly Rs. 40 crores. These institutions are IIT Bombay, IIT Delhi, IIT Kanpur, IIT Kharagpur, IIT Madras and IIT Ropar along with ARCI, Hyderabad, who contributed Rs. 2 crores each and BARC (supported through BRNS funding) contributed Rs. 3 crores. The remaining funds (Rs. 25 crores) came from Nano-mission of DST. Each partner institution has been provided with a workstation to operate the LEAP at IIT Madras remotely. These partner institutions send their samples to NFAPT, which are being prepared by Focused Ion Beam (FIB) and loaded on to the LEAP for remote operation by partner institutions. A designated scientist/technical staff of each partner institution has been trained on the remote operation of the LEAP, data acquisition and data analysis. Currently it is running round the clock with the help of three research engineers in 3 eight-hour shifts, coordinated by a research manager. Every partner institution is being provided with a time share of 8 hours per week (8 hours on FIB and 8 hours on LEAP). Beyond one slot per week, any researcher from partner institution can get excess slots at a charge of Rs. 30,000 per slot (each slot includes, 8 hours of FIB, 8 hours of LEAP usage and basic analysis of the results). The remaining machine time is distributed among other academic institutions, R&D laboratories and industries. Regular hands-on training and workshops are being conducted to generate competent human resource in the country with atomic level materials characterization capability. This unique facility would enhance the materials characterization capabilities of the whole nation in a big way. This is also expected to have a high impact on materials development in industries ranging from steel to automobiles and energy to transport sector.

National Facility for Atom Probe Tomography (NFAPT)

Investigators/Coordinators

Prof. B.S. Murty
Principal Investigator &
Principal Coordinator, IIT Madras

Prof. S. Sankaran
Principal Co-Investigator, IIT Madras

Dr. Ravi Sankar Kottada
Co-Investigator, IIT Madras

Dr. K.G. Pradeep
Co-Coordinator, IIT Madras

Dr. R. Gopalan
Co-Investigator and Coordinator, ARCI

Prof. Indradev S. Samajdar
Coordinator, IIT Bombay

Prof. Bodh Raj Mehta
Coordinator, IIT Delhi

Prof. Gouthama
Coordinator, IIT Kanpur

Prof. Rahul Mitra
Coordinator, IIT Kharagpur

Dr. Khushboo Rakha
Coordinator, IIT Ropar

Dr. Raghvendra Tewari
Coordinator, BARC, Mumbai

Research Manager/Engineers

Dr. M. Nagini
Research Manager, IIT Madras

Mr. Manu Mathai
Research Engineer, IIT Madras

Mr. N. Chandrasekaran
Research Engineer, IIT Madras

Mr. M. Srinivasan
Research Engineer, IIT Madras

Mr. Amit Vitthal Kumbhar
Research Engineer, IIT Bombay

Mr. Shivaprakash Solanki
Research Engineer, IIT Delhi

Dr. J. Bhagyaraj
Research Engineer, IIT Kanpur

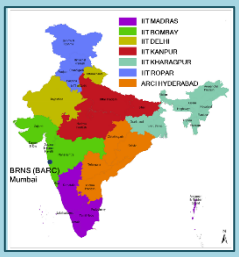
Mr. Palash Mukherjee
Research Engineer, IIT Kharagpur

Mr. Amit Kaushal
Research Engineer, IIT Ropar

Dr. Koppoju Suresh
Research Engineer, ARCI, Hyderabad

Dr. Amit Verma
Research Engineer, BARC, Mumbai

Partner Institutions and Respective Zones of NFAPT

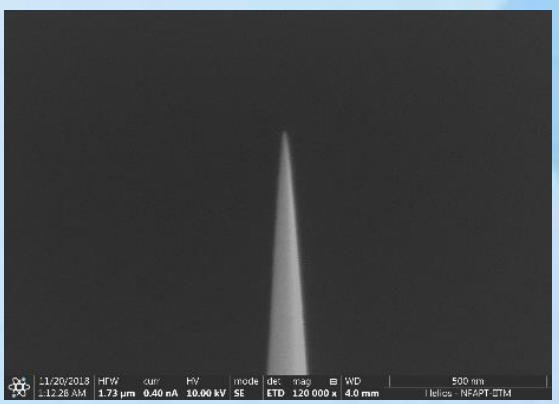
Name of the Coordinator	Partner Institution	States to be Taken care of by Partner Institution	Zone Map
Prof. Indradev S. Samajdar	IIT Bombay	Maharashtra, Gujarat and Goa	
Prof. Bodh Raj Mehta	IIT Delhi	Delhi, Rajasthan and Uttarakhand	
Prof. Gouthama	IIT Kanpur	Uttar Pradesh, Madhya Pradesh and Bihar	
Prof. Rahul Mitra	IIT Kharagpur	West Bengal, Jharkhand and North Eastern States	
Prof. B.S. Murty	IIT Madras	Tamil Nadu, Karnataka, Kerala, Pondicherry and other Union territories	
Dr. Khushboo Rakha	IIT Ropar	Punjab, Haryana, Himachal Pradesh and Jammu & Kashmir	
Dr. Raghavan Gopalan	ARCI, Hyderabad/Chennai	Andhra Pradesh, Telangana, Odisha and Chhattisgarh	
Dr. Raghavendra Tewari	BARC, Bombay	DAE laboratories	

NFAPT FACILITIES

Local Electrode Atom Probe (LEAP)

APT or 3-D Atom Probe is a high-performance microscope/spectroscope that provides a precise atomic information of a material, enabling a true 3-D atomic scale reconstruction, based on the principles of time of flight mass spectrometry and position sensitive detection. Due to its outstanding spatial resolution ($\Delta x \approx \Delta y \approx 0.3$-$0.5$ nm and $\Delta z \approx 0.1$-0.3 nm) and detection sensitivity, element concentrations down to a few ppm can be detected irrespective of elemental mass. LEAP 5000 XR has the capability to operate in voltage and laser modes depending upon the nature of the material and enables the investigation of a wide range of materials from metals, semiconductors to insulators/ceramics and polymers. This model provides high detection sensitivity and quality. The advanced pulsing module provides very high throughput.

LEAP 5000 XR at NFAPT, IIT Madras



APT specimen prepared using FIB

Dual Beam System (Helios G4 UX) at NFAPT, IIT Madras

TEM (Tecnai T12) at NFAPT, IIT Madras

Focused Ion Beam SEM and TEM

NFAPT has Dual Beam scanning electron microscope (FEI; Helios G4 UX SEM with Focused Ion Beam) for the preparation of APT specimens. Helios G4 UX incorporates state-of-the-art technologies that enable simple and consistent high-resolution S/TEM and APT sample preparation, as well as the highest-quality subsurface and 3-D characterization, even on the most challenging samples. It is equipped with a monochromater, EDS and EBSD facilities. Partner institutions send their samples to the NFAPT, which are being prepared using FIB and loaded in LEAP for remote access. Transmission electron Microscope (FEI; Tecnai T12 TEM) is also available at NFAPT for basic characterization of the samples prepared for LEAP through electropolishing technique.

Two-Day Workshop on Atom Probe Tomography, March 8-9, 2019, IIT Madras

A Two-Day Workshop on Atom Probe Tomography was conducted by NFAPT at IIT Madras during March 8-9, 2019. The workshop started with welcome address by Prof. Uday Chakkingal, Head, Department of MME, IIT Madras. Prof. B.S. Murty described about the NFAPT lab establishment and objectives of the workshop. This workshop was aimed to provide fundamentals as well as advanced knowledge of the APT and its applications. Considering that APT is a new technique in the domain of materials characterization in India, there is immediate need to establish a strong user base for optimal utilization of this national facility. A video clip about NFAPT and FIB sample preparation were screened on this occasion. The topics on APT, TEM Tomography, Nano SIMS and TOF SIMS were discussed during the workshop. Dr. K.G. Pradeep demonstrated the live remote mode operation of LEAP. Prof. B.S. Murty, IIT Madras; Mr. Peter Clifton, CAMECA Instruments, Madison; Dr. Chandan Srivastava, IISc, Bangalore; Dr. R. Balamuralikrishnan, DMRL, Hyderabad; Dr. R. Gopalan, ARCI, Chennai; Dr. K.G. Pradeep, IIT Madras; Dr. Deodatta Shinde, BARC, Mumbai; Dr. K.K. Marhas, Physical Research Laboratory, Ahmedabad; Dr. K.R. Vinoth Kumar, NCBS, Bangalore; Dr. Nirmalya Karar, NPL, Delhi and Dr. Krishanu Biswas, IIT Kanpur, delivered lectures. The talks were followed by FIB, LEAP, TEM lab visits at IIT Madras. This workshop provided a platform to the participants to familiarize on APT technique and motivated them to understand the importance of this in research and development and also in various other applications. This workshop was attended by 110 participants from 18 academic institutions, 4 R&D laboratories and 4 industries. The program was sponsored by Ametek India Pvt Ltd.

One-Day Workshop on Atom Probe Tomography, March 12, 2019, IIT Ropar

APT has been established at IIT Madras as a National Facility with IIT Ropar as one of the partner institutions. IIT Ropar is also responsible for the coordination of user proposals and to carry out measurements on samples from the Central Zone (states of Punjab, Haryana, Himachal Pradesh and Jammu & Kashmir). A One-Day Workshop on Advanced Characterization Symposium on 3-D Atom Probe Tomography was conducted by IIT Ropar on March 12, 2019. Inaugural remarks were given by Prof. Sarit K. Das, Director, IIT Delhi. This workshop was aimed to provide fundamentals as well as applications of APT. A video clip about NFAPT and FIB sample preparation were screened on this occasion. It was conducted by Dr. Khushboo Rakha, Coordinator of IIT Ropar. Prof. B.S. Murty, IIT Madras and Mr. Peter Clifton, CAMECA Instruments, Madison delivered lectures. The talks were followed by lab visit at IIT Ropar. This was the first seminar on APT at IIT Ropar and was attended by 50 participants.

Details of Workshops Conducted so far by NFAPT

Name of the Event	Place	Date
One-day workshop on Atom Probe Tomography	IIT Madras	January 9, 2012
One-day workshop on Atom Probe Tomography was conducted during EMSI-2017	IIT Madras	July 20, 2017
Demo was conducted for student participants of Prof. Brahm Prakash Memorial Materials Quiz (BPMMQ)	IIT Madras	August 5, 2017
Orientation program on APT	IIT Kanpur	October 7, 2017
Participants of Indo-Australian Workshop on Advances in Materials and Additive Manufacturing (AM^2) visited NFAPT	IIT Madras	March 22, 2018
Students from different schools visited NFAPT lab as a part of material camp conducted by ASM International, Chennai Chapter	IIT Madras	May 4, 2018
NFAPT Inauguration	IIT Madras	July 16, 2018
First lecture of NFAPT Lecture Series	IIT Madras	July 16, 2018
APT Sample Preparation Training	IIT Madras	July 25-27, 2018
Participants of Materials Characterization Certificate Course visited NFAPT	IIT Madras	July 28, 2018
Technical talk on Atom Probe Tomography and its applications-An Introduction	IIT Kanpur	July 28, 2018
One-day workshop on Atom Probe Tomography and its Applications	IIT Bombay	September 3, 2018
One-day workshop on Atom Probe Tomography	ARCI	September 7, 2018
LEAP/IVAS training	IIT Madras	October 25-26, 2018
One-day workshop on Atom Probe Tomography and its Applications	IIT Bombay	December 3, 2018
Participants of Advances in Nanotechnology Workshop visited NFAPT	IIT Madras	December 22-23, 2018
Two-day workshop on Atom Probe Tomography	IIT Madras	March 8-9, 2019
Advanced Characterization Symposium on 3-D Atom Probe Tomography	IIT Ropar	March 12, 2019

Visitors to NFAPT

Prof. K.S. Ravi Chandran, The University of Utah, USA, visited on January 2, 2019

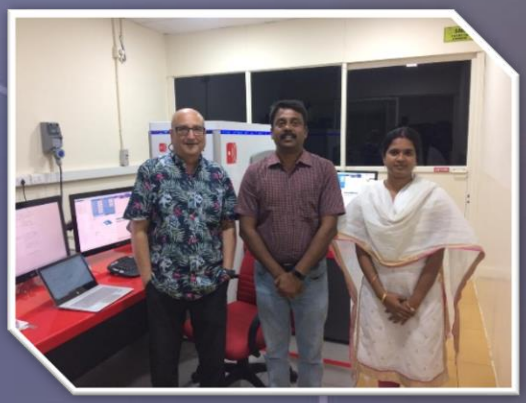
On the occasion of Institute Open House 2019 on January 3-4, 2019

Dr. Gurpreet Singh, Kanasa State University, USA, visited on January 7, 2019

IGCAR scientists visited on February 16, 2019

Sergiy V. Divinski, University of Munster, Germany, visited on February 19, 2019

On the occasion of Amalgam 2019 on March 2, 2019



Prof. Surendar Marya, Ecole Centrale De Nantes, France, visited on March 27, 2019

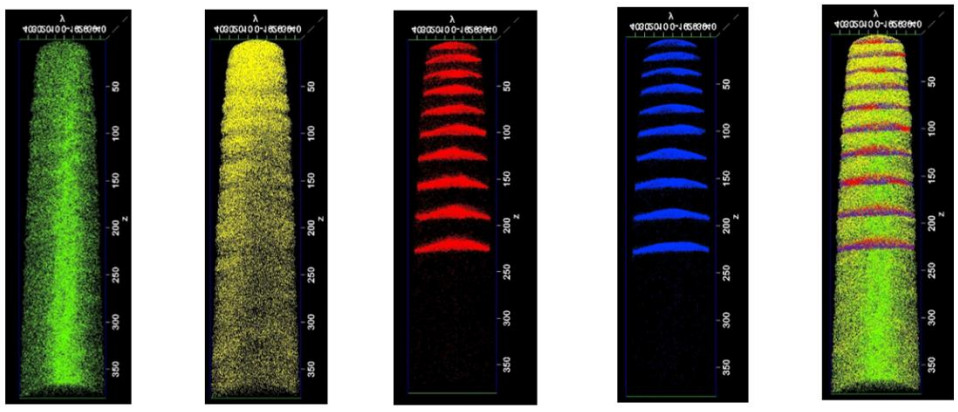
GE team (India and USA) visited on April 5, 2019

IIT Bombay

Material: *InAs/GaAs QDs (VCQDs)*

Uncoupled multilayered InAs/GaAs QD heterostructures with 50 nm GaAs layer thickness were grown on a semi insulating (100) GaAs wafers at a substrate temperature of 520 °C and a growth rate of 0.1 ML/s using a solid-source molecular-beam epitaxial system equipped with an effused material, such as Ga, In, Al, or Si, and an arsenic-valve cracker. At the same temperature, dots were overgrown with a combination capping of 3-nm quaternary In0.21Al0.21Ga0.58As and GaAs capping thicknesses of 50 nm. This layer was repeated for 10 periods, then capped with 100-nm GaAs capping at 590 °C. The total monolayer coverage for the InAs quantum dots was fixed at 2.7 ML at a growth rate of ~0.1 ML/s. 3-D reconstruction of InAs/InAl/GaAs shows the uniform distribution of QDs vertically.

Material: *InAs QD/InAl/GaAs*



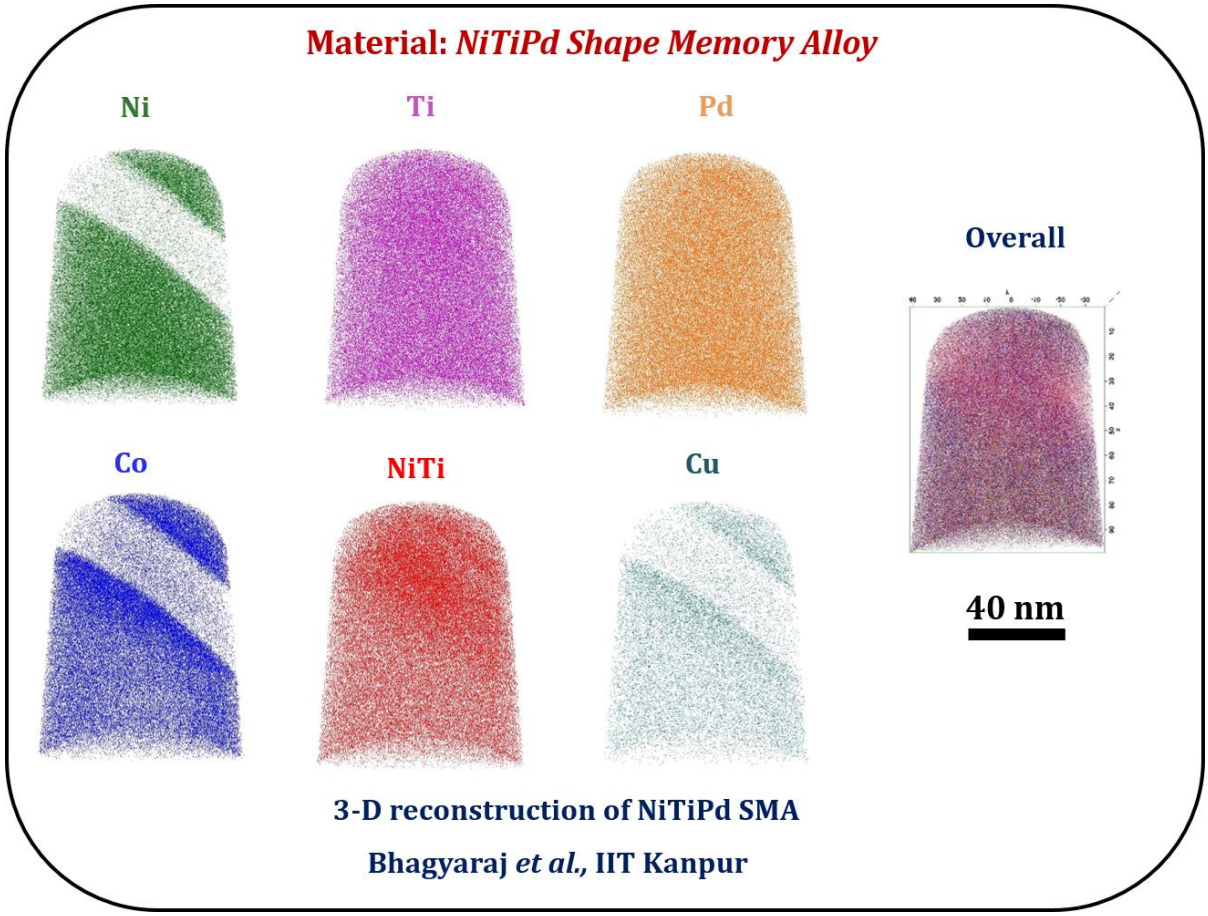
3-D reconstruction of InAs QD/InAl/GaAs layer

Binita Tongbram *et al.*, IIT Bombay

IIT Kanpur

Material: *NiTiPd Shape Memory Alloy*

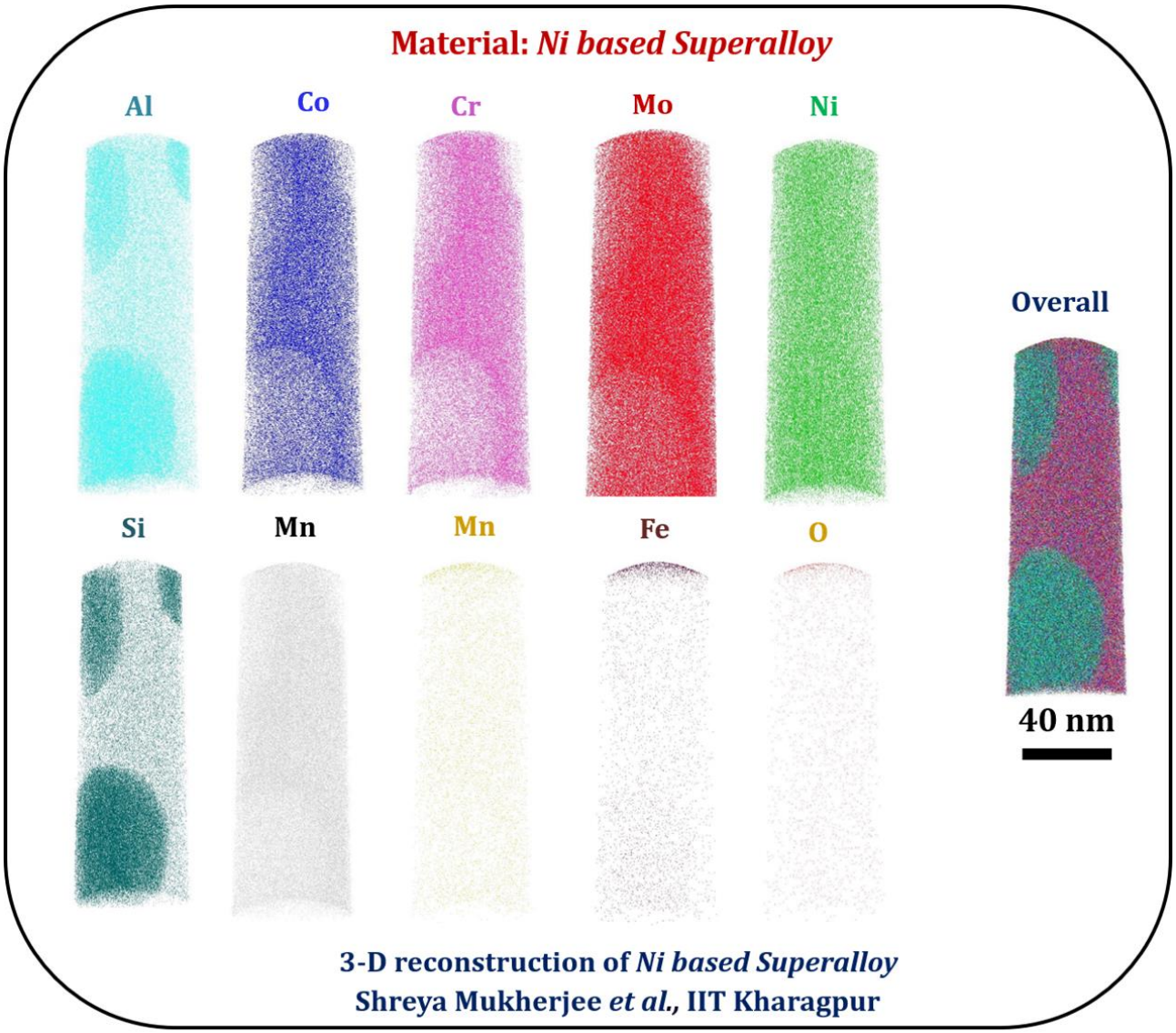
$Ni_{24.7}Ti_{50.3}Pd_{25.0}$ is high temperature shape memory alloy (SMA) with narrow thermal hysteresis and high thermal stability. The alloy was characterized in homogenized as well as in thermo-mechanically processed conditions for the occurrence of second phases, microstructural features, TTs and thermal cycling stability under stress-free conditions. Thermo-mechanical processed NiTiPd in thin strip form, was subjected to thermal cycling under load using a dynamic mechanical analyzer. Thermal cycling was carried out under a stress of 150 and 175 MPa for 2 heating-cooling cycles in the temperature range 50 to 250 °C and the functional response of the alloy was examined. The microstructural observations carried out using TEM have been correlated with the transformation characteristics of the alloy. The martensite phase with small twin width ratio was responsible for minimizing the interfacial energy and thereby resulting in the lower hysteresis of the alloys during phase transformation. Extensive efforts have been made in NiTiPd alloy to understand the microstructural aspects and transformation behavior. Microstructural study showed that the alloy has relatively higher volume fraction of Ti_2(Ni, Pd) precipitate phase. The reason for this observation is not clear at this moment and hence, further study is required on this aspect for resolving the issue. APT measurements were carried out to get the understanding of Interfaces analysis, precipitate/defect analysis and elemental distribution.



IIT Kharagpur

Material: *γ′ strengthened Ni based superalloy*

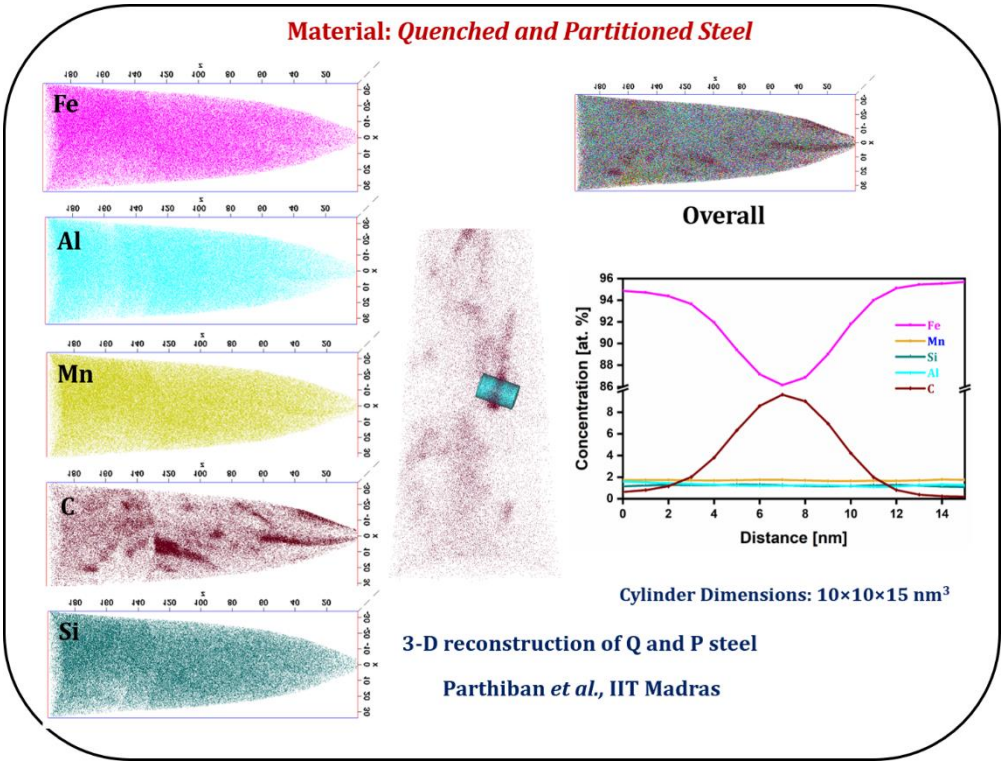
Haynes 282 is a new γ′-(Ni_3(Al, Ti)) strengthen Ni–based superalloy developed for high temperature structural applications, especially in aero and gas turbines. Haynes 282 is provided in the cast and forged condition. Two step treatment has been provided to put the alloy in high strength condition. Solution annealing was done in the range of 1121 to 1150 °C/1 h and then a prolong ageing treatment at 760 °C/220 h. The work aims to study the effect of ageing on the coarsening behavior of $L1_2$ ordered γ′ phase and the structure of γ/ γ′ interface. Overall the morphologies and mechanisms associated with particle coarsening upon aging have been a topic of significant discussion over the past decades. APT measurements were performed to study the coarsening behavior of γ′ precipitates and segregation of elements of between γ and γ′ phases.



IIT Madras

Material: *Quenched and Partitioned Steel*

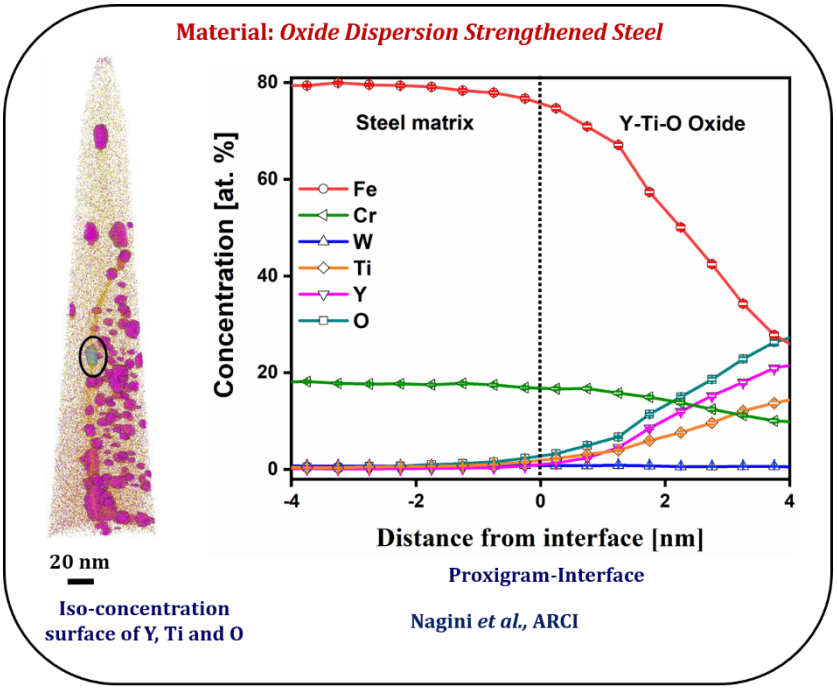
During the Q&P treatment the carbon from the supersaturated martensite partitions out and enriches the adjacent retained austenite. In the prior methods, the quantification of carbon content within the retained austenite was done by XRD method from the change in the 2θ position. It is an overall average estimation and does not throw any light on the nature of diffusion across a typical α'-γ grain boundary interface. APT studies were carried out for understanding the dynamics of the diffusion of the carbon atoms, showing the depleted zones and the enriched zones at the interface and also the characteristics of the interface in different strained conditions of the steel sample.



ARCI

Material: *Oxide Dispersion Strengthened (ODS) Steel*

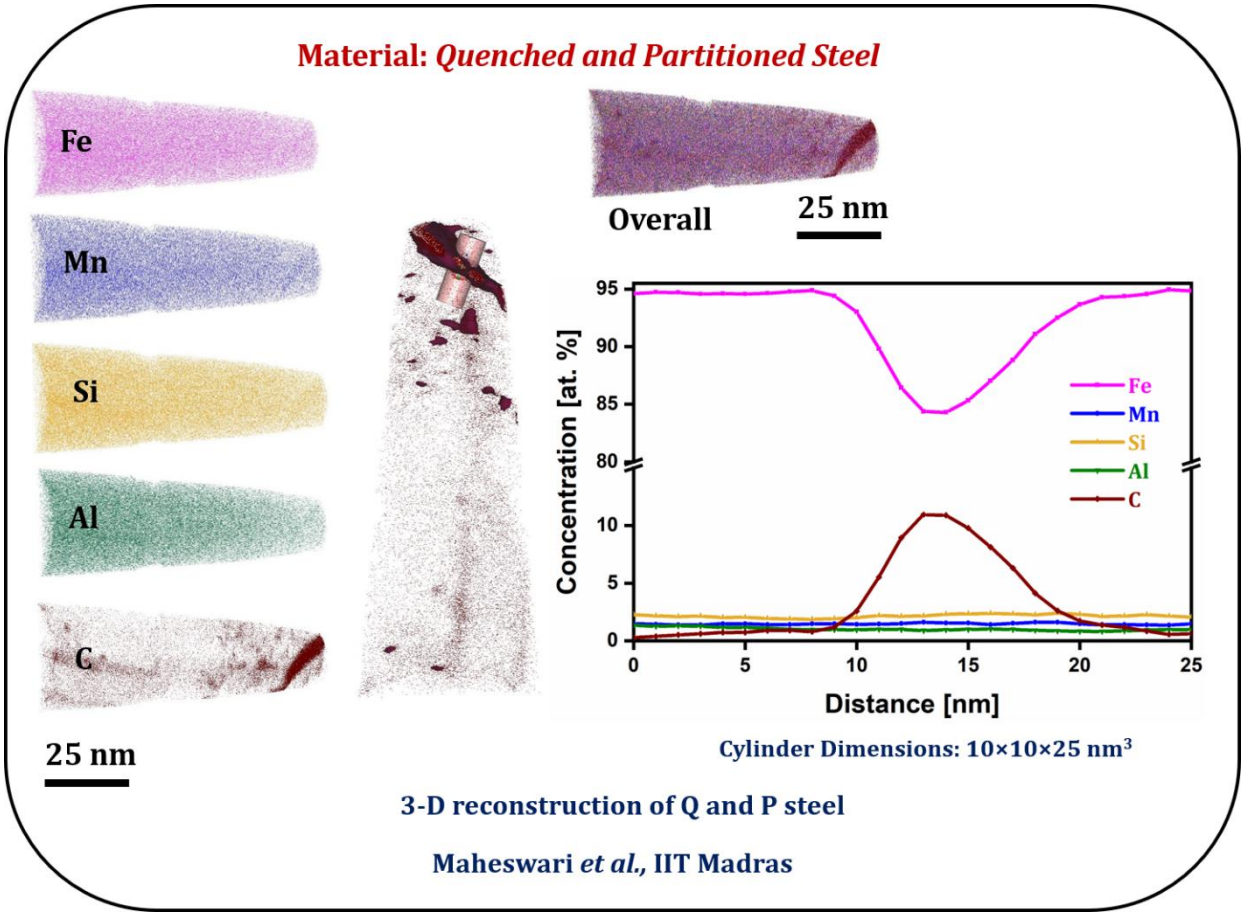
Oxide dispersion strengthened (ODS) ferritic 18Cr steels are being considered for high temperature structural applications such as, blankets for fusion reactors, fuel clads for Gen-IV fission reactors and blades for ultra-super critical steam & gas turbines due to their excellent combination of high temperature strength, creep strength and resistance to corrosion, oxidation and neutron irradiation. The dispersion of fine and thermally stable oxide particles (Y-Ti-O) in the ductile ferritic matrix results in pinning of fine-grained structure with oxide particles, which remarkably improves creep strength at elevated temperatures. APT results clearly showed extremely small Y-Ti-O oxide particles in ferritic matrix.



External Samples

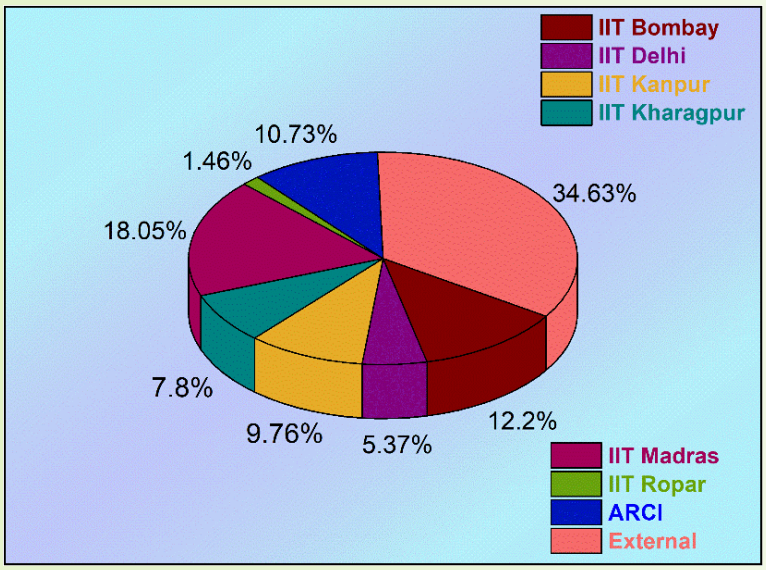
Material: *Quenched and Partitioned Steel*

The CMnSiAl-steel was subjected to quenching and partitioning process, for different partitioning temperature and time. The processed microstructure consists of lath martensite and retained austenite in form of grain as well as inter-lath. The retained austenite (γ) is present as individual grains and as inter-lath films between the lath martensite (M). Thickness of the martensite lath is estimated to be around 200 nm while the austenite film thickness is around 30-40 nm. Diffusion of carbon is expected to happen from martensite to retained austenite phase through the interface during the partitioning process. Thus, quantification of carbon in both phase and at the interface are mandatory to prove the mechanism of Q&P process. Also, it is important to find that whether local segregation (in nm) of any other alloy element is present or not. XRD and EPMA studies couldn't solve these purposes. APT is the only possible technique as far now, to quantify the carbon content in nanometer range. Thus, it helps to understand the Q&P process and to propel our current research field. APT measurements were carried out to estimate the elemental distribution and quantification of Fe, C, Mn, Si and Al across the M- γ interface.



LEAP Slot Statistics

August 2018-March 2019	
Partner Institution	**No. of Slots Used**
IIT Bombay	25
IIT Delhi	11
IIT Kanpur	20
IIT Kharagpur	16
IIT Madras	37
IIT Ropar	03
ARCI	22
External	71
Total	**205**



Journal Publications of NFAPT

1. Karati, A., Nagini, M., Ghosh, S., Shabadi, R., Pradeep, K.G., Mallik, R.C., Murty, B.S. and Varadaraju, U.V., Ti2NiCoSnSb-A New Half-Heusler Type High-Entropy Alloy Showing Simultaneous Increase in Seebeck Coefficient and Electrical Conductivity for Thermoelectric Applications, Sci. Rep., 9:5331 (2019) 1-12.
2. M. Nagini, K.G. Pradeep, R. Vijay, A.V. Reddy, B.S. Murty and G. Sundararajan, A Comprehensive Characterization of Dispersoids in Oxide Dispersion Strengthened 18Cr Ferritic Steel Using Electron Microscopy, Atom Probe Tomography and Small Angle X–ray Scattering study, to be communicated.

Nature of Samples Studied so far at NFAPT

- High Entropy Alloys
- Oxide Dispersion Strengthened Steels
- Interstitial Free (IF) Steels
- MgO Composite Materials
- Bainitic Steels
- Quenched and Partitioned Steels
- Superalloys
- Vertically Coupled Multilayered InAs/GaAs QDs
- Magnetic Materials
- $CoSb_3$ Skutterudites
- CuZnS Thin Films
- NiTi and NiTiPd Shape Memory Alloys
- Zno/NiO Heterointerfaces
- LAT 971 Composites
- W and W-Ti based composites
- Co-Sm Magnetic Materials
- Bulk Metallic Glasses

National Facility for Atom Probe Tomography (NFAPT)

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Research Proposal

1. Name of the project investigator and address:

   Name of the student involved in this work and mobile number:

2. Name and number of the samples:

3. Briefly describe fabrication method and nature of sample: *(max. 50 words)*
   (Metals, semiconductors, ceramics, composites, nanowires, etc.)

4. Nature of sample:

• Is it magnetic? Yes/No	• Is it electron beam sensitive? Yes/No
• Is it volatile? Yes/No	• Is it flammable? Yes/No

5. Motivation for using atom probe tomography, in comparison to other characterization tools: *(max. 100 words)*

6. Nature of work: *(max. 50 words)*
   (Grain boundary/phase boundary/interface analysis, cluster analysis, dopant mapping, etc.)

7. Is the atom probe sample already prepared?

   - *If Yes, please describe the details of preparation method and sample dimensions (max. 50 words)*
   - *If No, the type of sample preparation needed. (Electropolishing or Focused Ion Beam)*

8. Is site specific sample preparation mandatory? If so, mention the specific area in the sample.

Note:

- Please share results that may be helpful for APT sample preparation and APT analysis (*SEM (mandatory)and TEM micrographs*)
- Please list one or two APT publications related to this work, if available
- **Dimensions of the samples sent to IITM should be less than 10×10×5 mm and in well-polished condition, e.g., suitable for EBSD.**
- Please send the filled form to the respective zone coordinator to get APT slot. For more details please visit nfapt.iitm.ac.in

National Facility for Atom Probe Tomography (NFAPT)

Helios G4 UX

LEAP 5000 XR

Tecnai T12

How to use LEAP@NFAPT:

Anyone interested to use LEAP at NFAPT needs to fill up a proposal document that is available on the NFAPT website and submit the proposal to either the Coordinator of his/her region (https://nfapt.iitm.ac.in/contact.html) or to Prof. B.S. Murty (murty@iitm.ac.in). Once the proposal is accepted, the Research Manager, NFAPT will provide the slot and the samples can be sent to IITM for FIB sample preparation and LEAP study and analysis.

Slots at NFAPT:

Each slot at NFAPT consists of 8 hours of sample preparation on FIB and 8 hours of characterization of samples on LEAP. The slot also includes basic reconstruction and analysis of the data obtained by the LEAP. The number of samples that can be studied in each slot depends on the nature of sample.

NFAPT usage charges:

Academic Institutions and Govt. R&D labs - Rs. 30,000 per slot (Please note that each slot includes 8 hours of FIB and 8 hours of LEAP and about 2-4 hours of reconstruction and analysis of the data obtained. This is a highly subsidized charge for such a sophisticated analytical facility).

Industries - Rs. 1,50,000 per slot.

For any details about NFAPT please contact:
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NFAPT welcomes APT proposals from

Academic Institutions,

R&D Laboratories and Industries.