

NFAPT Third Newsletter of National Facility for Atom Probe Tomography

Issue 3

August 2019



LEAP 5000 XR



Duel Beam System (Helios G4 UX)



TEM (Tecnai T12)









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About NFAPT

To improve the quality of materials research and development in the country with the help of atomic level characterization, National Facility for Atom Probe Tomography (NFAPT) has been set up at IIT Madras with seven partner institutions, namely, IIT Bombay, IIT Delhi, IIT Kanpur, IIT Kharagpur, IIT Madras, IIT Ropar, ARCI Hyderabad, who contributed 35% of the cost of the facility and DST Nano Mission providing the remaining 65%. The NFAPT was inaugurated by Prof. Ashutosh Sharma, Secretary, DST and Prof. Bhaskar Ramamurthi, Director, IIT Madras on July 16, 2018. NFAPT houses the state-of-the-art LEAP (Cameca, LEAP 5000 XR) along with Focused Ion Beam (FEI, Helios G4 UX FIB) for LEAP sample preparation and a Transmission Electron Microscope (FEI, Tecnai T12 TEM). This is the first remotely operable material characterization facility of the country and the first remotely operable LEAP in the world. Each partner institution has been provided with a workstation to operate the LEAP at IIT Madras remotely.

The partner institutions send their samples to NFAPT, which are being prepared by FIB and loaded on to the LEAP at IITM 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 NFAPT is being run 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). The remaining machine time is distributed among other academic institutions, R&D laboratories and industries in the country. 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.

Research Manager/Engineers

Investigators/Coordinators

Prof. B.S. Murty Prof. Gouthama Dr. M. Nagini Mr. Shivaprakash Solanki Principal Investigator and Coordinator **Research Manager Research Engineer Principal Coordinator** IIT Kanpur **IIT Madras IIT Delhi IIT Madras** Prof. Gouthama Prof. S. Sankaran Prof. Rahul Mitra Mr. Manu Mathai **Coordinator** and Principal Co-Investigator Coordinator **Research Engineer Research Engineer IIT Madras IIT Kharagpur IIT Madras IIT Kanpur** Dr. Ravi Sankar Kottada Dr. Khushboo Rakha Mr. N. Chandrasekaran Mr. Palash Mukherjee **Co-Investigator** Coordinator **Research Engineer Research Engineer IIT Madras** IIT Ropar **IIT Madras IIT Kharagpur** Dr. R. Gopalan Dr. Raghavendra Tewari Mr. M. Srinivasan Mr. Amit Kaushal **Co-Investigator** and Coordinator **Research Engineer Research Engineer** Coordinator **DAE** laboratories **IIT Madras** IIT Ropar ARCI **Prof. Anil Kottantharavil** Dr. K.G. Pradeep Dr. Koppoju Suresh Dr. Amit Verma Coordinator **Co-Coordinator Research Engineer Research Engineer IIT Bombay IIT Madras** ARCI. Hyderabad BARC, Mumbai Prof. Ankur Goswami **Dr. Sumantra Mandal** Mr. Amit Vitthal Kumbhar Coordinator Co-Coordinator **Research Engineer IIT Delhi** IIT Kharagpur **IIT Bombay**

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. Ankur Goswami	IIT Delhi	Delhi, Rajasthan and Uttarakhand	HT BOMBAY HT DELHI HT KANDID
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	BRINS (BARE)
Dr. Khushboo Rakha	IIT Ropar	Punjab, Haryana, Himachal Pradesh and Jammu & Kashmir	Mumbai Vica and State
Dr. Raghavan Gopalan	ARCI, Hyderabad/Chennai	Andhra Pradesh, Telangana, Odisha and Chhattisgarh	
Dr. Raghavendra Tewari	BARC, Bombay	DAE laboratories	A g

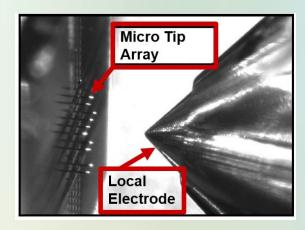
NFAPT FACILITIES

Local Electrode Atom Probe (LEAP)

APT or 3-D Atom Probe is а 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





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



Focused lon 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 highestquality 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.

TEM (Tecnai T12) at NFAPT, IIT Madras

Workshop on Advances in Nano-Scale Materials Characterization, July 7, 2019, IIT Madras

A One-Day Workshop (ISMANAM-2019 Pre-Conference workshop) on "Advances in Nano-Scale Materials Characterization" was conducted by NFAPT at IIT Madras on July 7, 2019. The workshop started with welcome address by Prof. B.S. Murty, Department of MME, IIT Madras. The workshop is aimed to provide fundamental aspects of nano-scale materials characterization using advanced microscopy techniques at near-atomic resolutions including aberration corrected TEM and APT and their collective applications. Dr. Christian Liebscher, Max-Planck-Institut für Eisenforschung, Germany; Prof. Christian Kübel Karlsruhe Institute of Technology (KIT) and Technische Universität (TU), Darmstadt, Germany; Prof. Paul Voyles University of Wisconsin-Madison, USA; Dr. Peter Clifton Cameca Inc., USA; Prof. Simon Ringer, The University of Sydney, Australia delivered lectures. The talks were followed by FIB and LEAP lab visits at IIT Madras. The details of the speakers and titles are mentioned below. This workshop provided a platform to the participants to familiarize on in Nano-Scale Materials Characterization and motivated them to understand the importance of this in research and development and also in various other applications. This workshop was attended by 80 participants from 9 academic institutions and 2 R&D laboratories. The program was sponsored by Ametek India Pvt Ltd. It was conducted by Dr. K.G. Pradeep of Department of MME, IIT Madras and NFAPT Team.

S. No.	Speaker Name	Title of the Talk
1.	Dr. Christian Liebscher Max-Planck-Institut für Eisenforschung (MPIE), GmbH, Düsseldorf, Germany	Introduction to conventional and aberration corrected transmission electron microscopy
2.	Prof. Christian Kübel Karlsruhe Institute of Technology (KIT) and Technische Universität (TU), Darmstadt, Germany	Analytical transmission electron microscopy and introduction to in- situ techniques as well as electron tomography
3.	Prof. Paul Voyles University of Wisconsin-Madison, USA	Solving structurally complex materials
4.	Dr. Peter Clifton Cameca Inc., USA	Introduction to atom probe tomography and current trends
5.	Prof. Simon Ringer The University of Sydney, Australia	Advances in atom probe microscopy and intersections with first principles modelling



One-Day Workshop on Atom Probe Tomography, July 10, 2019 at IIT Kharagpur

APT has been established at IIT Madras as a National Facility with IIT Kharagpur as one of the partner institutions. IIT Kharagpur is also responsible for the coordination of user proposals and to carry out measurements on samples from the Central Zone (states of Uttar Pradesh, Madhya Pradesh and Bihar). A One-Day Workshop on "Redefining the Horizons of Nanoscale Composition Characterization-Laser assisted Atom Probe Tomography" was conducted by IIT Kharagpur on July 10, 2019. This workshop was aimed to provide fundamentals as well as applications of APT. It was conducted by Dr. Rahul Mitra, Coordinator of IIT Kharagpur. Mr. Peter Clifton, CAMECA Instruments, Madison delivered lecture. The talk was followed by IVAS training session for selected research scholars at IIT Kharagpur. This was the first seminar on APT at IIT Kharagpur and was attended by 70 participants.



First Anniversary of NFAPT, July 16, 2019 at IIT Madras

The unique National Facility of Atom Probe Tomography (NFAPT) was inaugurated on July 16, 2018 at IIT Madras. To mark this day of establishment of NFAPT, the First Anniversary of NFAPT was celebrated on July 16, 2019 at IIT Madras. Coordinators of partner institutions of NFAPT, faculty members from various materials research departments of IIT Madras were present on the occasion. Dr. K. Madangopal Krishnan, Associate Director, Materials Group, BARC, was the Chief Guest of the program. Prof. B.S. Murty described about the NFAPT lab establishment. A video clip about NFAPT was screened on this occasion. Dr. K.G. Pradeep, Co-Coordinator of IIT Madras presented the activities of NFAPT since its Inauguration. Remarks on behalf of the partner institutions were given by Dr. R. Gopalan, Coordinator of ARCI and Prof. Gouthama, Coordinator of IIT Kanpur. Prof. Ravindra Gettu, Dean (IC&SR), IIT Madras appreciated the facility and wished the NFAPT the very best. Dr. M.K. Sapra, Head & Scientific Secretary, BRNS, Bombay was shared his valuable remarks on this occasion. The Chief Guest of the function explained the importance of collaborative science infrastructure. Dr. K.G. Pradeep demonstrated the live remote mode operation of LEAP. As originally planned, NFAPT is running is 3 shifts every day on a 24x6 basis and has helped a large number of materials scientists throughout the country and a few overseas to understand a variety of materials at the atomic level.



Details of Workshops Conducted so far by NFAPT

S. No	Name of the Event	Place	Date
1	One-day workshop on Atom Probe Tomography	IIT Madras	January 9, 2012
2	One-day workshop on Atom Probe Tomography was conducted during EMSI-2017	IIT Madras	July 20, 2017
3	Orientation program on APT	IIT Kanpur	October 7, 2017
4	NFAPT Inauguration	IIT Madras	July 16, 2018
5	First lecture of NFAPT Lecture Series	IIT Madras	July 16, 2018
6	APT Sample Preparation Training	IIT Madras	July 25-27, 2018
7	Technical talk on Atom Probe Tomography and its applications-An Introduction	IIT Kanpur	July 28, 2018
8	One-day workshop on Atom Probe Tomography and its Applications	IIT Bombay	September 3, 2018
9	One-day workshop on Atom Probe Tomography	ARCI	September 7, 2018
10	LEAP/IVAS training	IIT Madras	October 25-26, 2018
11	One-day workshop on Atom Probe Tomography and its Applications	IIT Bombay	December 3, 2018
12	Two-day workshop on Atom Probe Tomography	IIT Madras	March 8-9, 2019
13	Advanced Characterization Symposium on 3-D Atom Probe Tomography	IIT Ropar	March 12, 2019
14	ISMANAM-2019 Pre-Conference Workshop on Advances in Nano-Scale Materials Characterization	IIT Madras	July 7, 2019
15	One-Day Workshop on "Redefining the Horizons of Nanoscale Composition Characterization-Laser assisted Atom Probe Tomography"	IIT Kharagpur	July 10, 2019

NFAPT Newsletter



Students of ASM Materials camp visited NFAPT on May 13, 2019



GE Bangalore team visited on May 28, 2019



On the occasion of ISMANAM 2019 on July 12, 2019



On the occasion of ISMANAM 2019 on July 13, 2019



Dr. Kakodkar, Former Chairman, DAE visited LEAP facility on Aug 3, 2019



Prof. M.S. Ananth, Former Director, IIT Madras visited on August 5, 2019

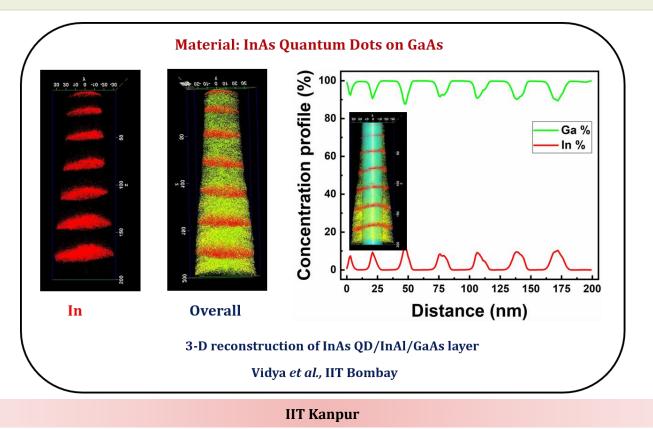


Prof. Rajan Ambat, Technical University Denmark visited on August 13, 2019

IIT Bombay

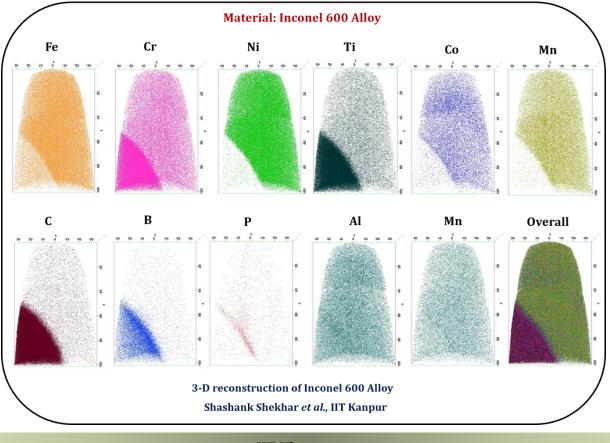
Material: InAs/GaAs QDs (VCQDs)

The active layer of the heterostructure is a 10-layered InAs quantum dots with successive capping of 60nm GaAs. From cross-sectional HRTEM analysis, it was found that the shape of InAs quantum dots were tetrahedral or truncated pyramids. The average size of these QDs were found to be 17 nm (bottom base), 12 nm (top base), and 2 nm (QD height). The main motive for using atom probe tomography was to find out the indium concentration in each InAs QDs in the ten- layered heterostructure along the growth direction. The distribution of In (red), Ga (Green) and As (yellow) in the QD and capping layer is analyzed using Atom probe tomography and the 3D reconstruction images are shown in Figure. A cylindrical region of interest was considered in the heterostructure across the dot layers to calculate concentration profiles for In and Ga in order to investigate if there is any indium out-diffusion from the dots to the capping layer. The interface analysis of InAs QD/ GaAs for each layer was done and the concentration profiles (%) obtained for In and Ga.



Material: Inconel 600 alloy

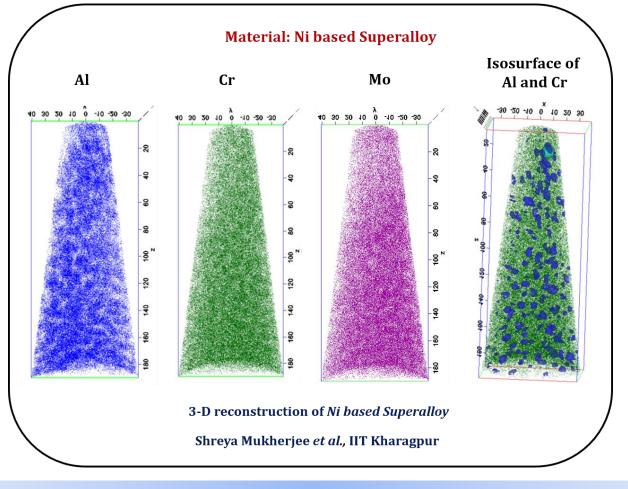
Segregation at grain boundaries are known to be deleterious to their overall mechanical and functional properties. Moreover, segregation of carbon leads to formation of carbide phases which are even more deleterious. Extent of segregation and precipitation depends on grain boundary energy and hence its character. However, no direct evidence has been shown which correlates grain boundary character with segregation and precipitation. APT measurements were carried out to get the understanding of grain boundary analysis, precipitate/defect analysis and segregation analysis.



IIT Kharagpur

Material: γ' strengthened Ni based superalloy

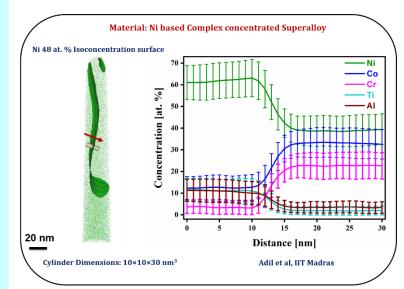
Haynes 282 is a new γ' -(Ni₃(Al, Ti)) strengthen Ni–based superalloy developed for high temperature structural applications, especially in aero and gas turbines. Haynes 282 is provided in the cast and forged condition. Two step treatment has been provided to put the alloy in high strength condition. Solution annealing was done in the range of 1121 to 1150 °C/1 h and then a prolong ageing treatment at 650 °C/24 h. The work aims to study the effect of aging on the coarsening behavior of L1₂ 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: Ni based Complex Concentrated SuperAlloy

Ni-based superalloys are often used in gas turbines and plane engines, due to their high-temperature mechanical strength. Interfaces play a critical role at those operating temperatures in these alloys. These alloys have a characteristic γ/γ' microstructures with a low interface energy, coherent interfaces between a solid solution face-centered cubic (FCC)- γ matrix phase and block-shaped L1₂ ordered γ' precipitates. The elastic misfit stresses across the γ/γ' interface often controls their high temperature mechanical and coarsening behaviour. Atomic scale composition analysis was carried out across the γ/γ' interface using atom probe tomography (APT).



IIT Ropar

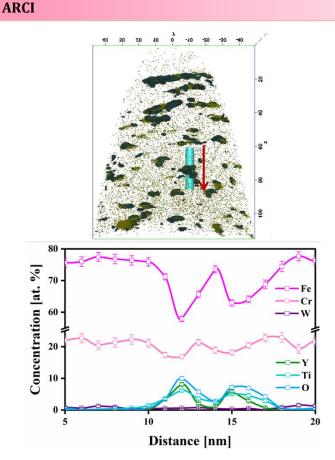
Material: Bainitic Steel

The APT measurements were carried out on nanostrucutred bainitic steel to examine the distribution of carbon after ausforming. All elements are uniformly distributed in nano bainitic steel except carbon. The analysis shows that while the distribution of substitutional alloying elements does not change subsequent to thermomechanical treatment, there is a large effect on carbon redistribution. While nanostructured bainite formed isothermally and without prior deformation showed two populations of highly enriched austenite phase and low carbon bainite phase, after ausforming with a strain of 15% the redistribution has changed. We can see segregation of carbon which is believed to be on dislocation pile ups and twins formed during ausforming.



Material: Oxide Dispersion Strengthened (ODS) Steel

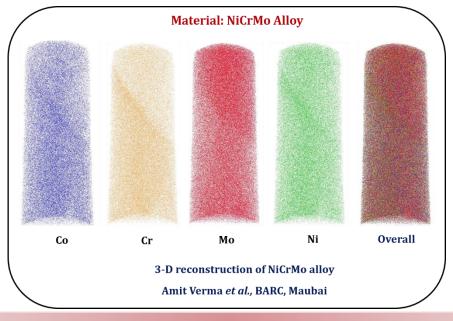
Oxide dispersion strengthened (ODS) ferritic 18Cr steels are being considered for high temperature structural applications such as, blankets for fusion reactors, fuel clads for Gen-IV fission reactors and blades for ultra-super critical steam & gas turbines due to their excellent combination of high 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 strength elevated improves creep at temperatures. APT results clearly showed extremely small Y-Ti-O oxide particles in ferritic matrix.

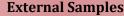


DAE Laboratories

Material: NiCrMo Alloy

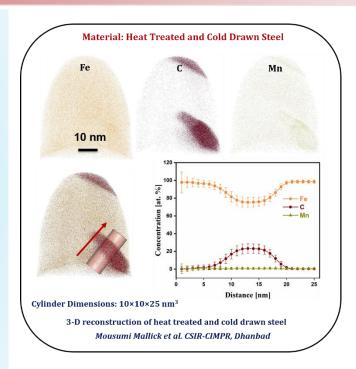
Ni-Cr-Mo based alloys have also been considered as candidate materials for long-term storage applications of radioactive waste up to a period of 10,000 years in geological repository sites at temperatures varying in the range of 100-300 °C. However, the prolonged aging during service of these alloys in the temperature range of 400-700 °C results in the precipitation of few nanometer sizes precipitates of intermetallic Ni₂(Cr,Mo) phase (which has Pt₂Mo type prototype oI6 structure) from the disordered face-centered cubic (fcc) matrix. Precipitation of Ni₂(Cr,Mo) phase deteriorates mechanical and corrosion properties significantly. An understanding of the chemistry of the nanometer sized precipitates of Ni₂(Cr,Mo) phase is important as composition of alloys could be fine-tuned to retard and/or suppress this precipitation reaction. In order to determine compositions of the nano precipitates of Ni₂(Cr,Mo) phase, APT examination was carried out on Ni-Cr-Mo alloy.



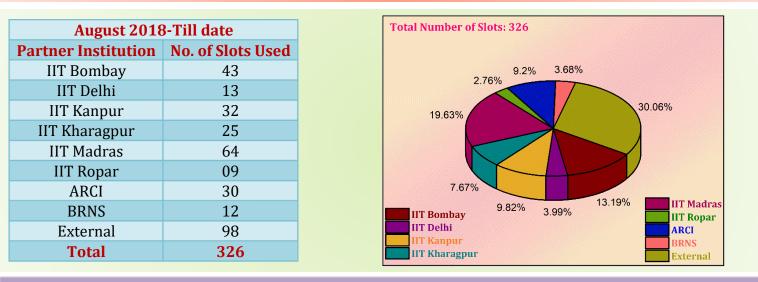


Material: Heat Treated and Cold Drawn Steel

The understanding of atomic phenomena especially local phase transformation is essential for the material property. Further to have an idea of full understanding of the real material at atomic scale in three-dimension. The elemental distribution of the material and position of each atom of the undergoing study at the atomic scale is very much essential for the present study. APT was carried out for the mapping of atoms of different elements (Mainly, iron, carbon and manganese) at preferred location of the most significant microstructural features; namely, iron-based matrix and spheroid precipitation (sub-microscopic size) so as to identify the elemental distribution of both the phases.



LEAP Slot Statistics



Journal Publications of NFAPT

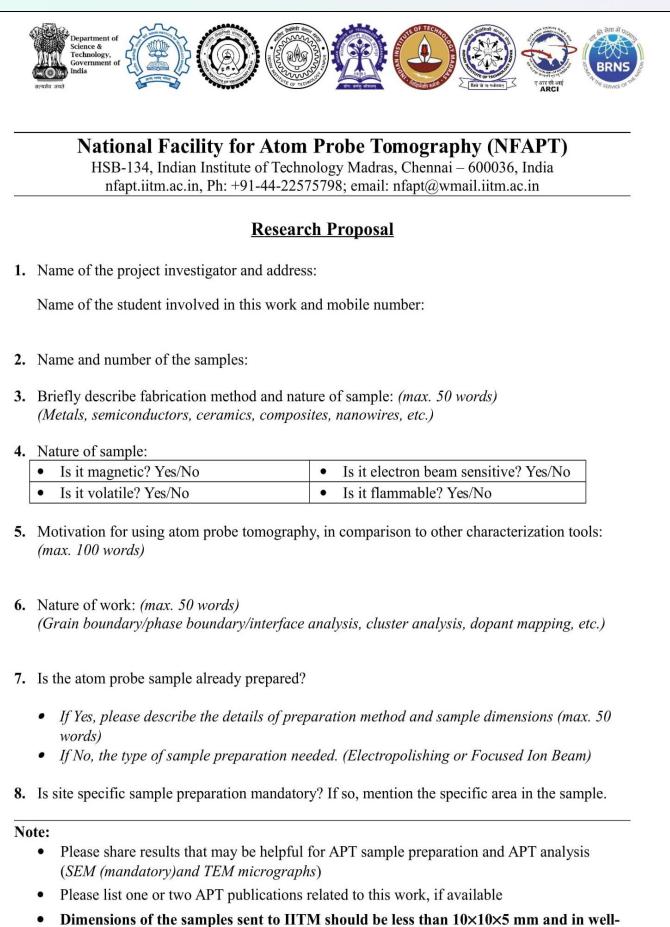
- Karati, A., Nagini, M., Ghosh, S., Shabadi, R., Pradeep, K.G., Mallik, R.C., Murty, B.S. and Varadaraju, U.V., Ti₂NiCoSnSb-A New Half-Heusler Type High-Entropy Alloy Showing Simultaneous Increase in Seebeck Coefficient and Electrical Conductivity for Thermoelectric Applications, Sci. Rep., 9:5331 (2019) 1-12.
- 2. Sowjanya. M and Kiran S. R. N.M, In-situ high temperature micro-Raman investigation of annealing behavior of high-pressure phases of Si, J. Appl. Phys. 125 (2019) 225105.
- 3. 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, Submitted to Journal of Alloys and Compounds.
- 4. Shaik Adil, M.V. Suraj, Lava Kumar Pillari, Soumya Sridar and B.S. Murty, Role of Fe in A₃B ordered intermetallic phase in Al-Co-Cr-Ni-Ti high-entropy alloy and its influence on the microstructure, Communicated to Acta Mater., 2019.
- 5. Karati, A., Hariharan, V.S, A. Prasad, S. Ghosh, R.C. Mallik, R. Shabadi, L Bichler, B.S. Murty and U.V. Varadaraju, Synthesis and thermoelectric properties of high entropy Half-Heusler Alloys. To be Communicated.
- 6. Mayur Vaidya, Karati, A, Guru Vidyathri, K.G. Pradeep, B.S. Murty, Synthesis and microstructural studies of a new senary high entropy alloys. To be Communicated.

Nature of Samples Studied so far at NFAPT

4	High	Entropy	Alloys
	піgn	Entropy	Alloys

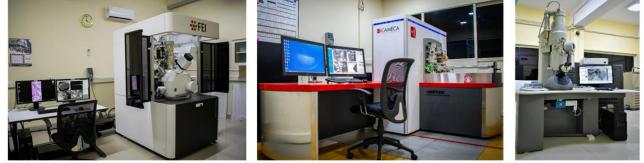
- Oxide Dispersion Strengthened Steels
- Interstitial Free (IF) Steels
- MgO Composite Materials
- Bainitic Steels
- Quenched and Partitioned Steels
- Superalloys
- Vertically Coupled Multilayered InAs/GaAs QDs
- Magnetic Materials
- CoSb₃ Skutterudites
- CuZnS Thin Films
- 🔱 NiTi and NiTiPd Shape Memory Alloys

- LAT 971 Composites
- 4 Zno/NiO Heterointerfaces
- W and W-Ti based composites
- Co-Sm Magnetic Materials
- Bulk Metallic Glasses
- Ni electroplated interfaces (Cu-Ni, Cu-Steel)
- NiCrMo Alloy
- Nobel Metal Clusters
- High Carbon Steel
- Ni-Zr-Al
- 9Cr-1Mo Steel



- polished condition, e.g., suitable for EBSD.
 Please send the filled form to the respective zone coordinator to get APT slot. For more
- Please send the filled form to the respective zone coordinator to get APT slot. For more details please visit nfapt.iitm.ac.in





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: Rs. 40,000 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).

Govt. R&D labs: Rs. 1,00,000 per slot Industries: Rs. 1.50.000 per slot.

> For any details about NFAPT please contact: **Research Manager** National Facility for Atom Probe Tomography Indian Institute of Technology Madras Chennai-600036, Tamil Nadu, India Phone: +91-44-22575798 Mail: nfapt@wmail.iitm.ac.in Web: www.nfapt.iitm.ac.in

NFAPT welcomes APT proposals from Academic Institutions, **R&D** Laboratories and Industries.