Establishing Atom Probe Tomography (APT) at KTH

– 3D mapping of materials structures with sub-nanometer spatial resolution

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January 2021

1 Summary

The Hultgren Laboratory is a part of the School of Industrial Engineering and Management (ITM) and one of twelve strategic research and innovation infrastructure facilities at KTH, located at the Department of Materials Science and Engineering (MSE). The Hultgren Laboratory infrastructure is an open meeting place for academia, industry, and other actors in society to develop competence and create new synergies for the joint effort to solve scientific challenges.

The Hultgren Laboratory supports researchers by carrying out structural analysis of materials for energy, automotive, aviation, construction, and medical applications. Electron microscopy techniques have been key for this work. However, the scope of such techniques is restricted for sub-nanometer characterization of materials. Therefore, there is a growing need for the atom probe tomography (APT) technique, which enables capturing 3D atomic composition maps of materials structures.

APT equipment is nowadays an effective laboratory tool, capable of meeting the needs of a multi-user laboratory. We have experienced a consistent increase in the need to access this type of advanced measurement in our research, while the only current APT in Sweden is now 14 years old and approaching the end of its technological lifetime. Thus, we foresee the need to increase the capacity and access to these types of measurements in Sweden. This is important for the competitiveness of our materials-related research.

The purpose of this briefing is therefore to inform you about the ongoing effort to equip KTH with state-of-the-art APT equipment. We hope to bring awareness about the potential impact this would have on KTH's research and education in materials science and related fields. We hope this investment would decrease the barrier of entry for academic and industrial users to access advanced materials characterization tools to benefit society.

2 Background

APT has opened new opportunities for researchers to develop new insight and understanding of materials nanostructures which has not been explored fully before. The new information is in 3D at the atomic scale, an example of the 3D material information of a maraging steel obtained from APT is shown in Figure 1. APT captures 3D chemical composition maps of materials at sub-nanometer spatial resolution (~0.1 nm) with outstanding chemical detection sensitivity (~ 5 ppm). This 3D composition mapping provides pivotal information about structures governing materials properties, for example, nanostructures, phase interphases, precipitates, grain boundaries, clusters, and chemical decoration of defects within the material.

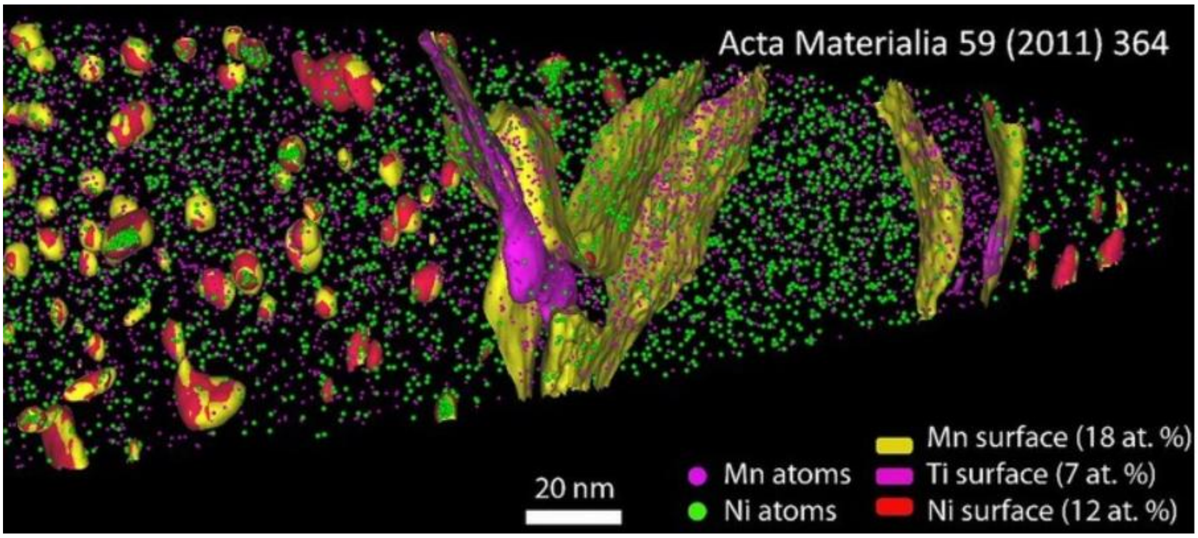

Figure 1. *Chemical gradients across phase boundaries of martensite and austenite phases in maraging steel obtained by APT. Courtesy Prof. Dierk Raabe, Max-Planck Institute, Düsseldorf.*

Especially after a new commercial version of the APT equipment became available in 2005, it has had a massive impact on materials research reflected by a five-fold increase in the number of publications per year as of 2019. The importance of APT has been recognized globally. At the same time, Swedish APT research capacity continues to fall relative to others (no.17 in 2019), although Chalmers University in Sweden is recognized for having leading instrument expertise. Historically, APT has been especially attractive in the field of metallurgy due to excellent results and high specimen yield but since the introduction of laser-assisted evaporation, the range of materials applications has broadened. Together with the technological advancement during the last decade, this has opened up new opportunities to use APT measurements in the fields of, for example, semiconductor devices, biology[1] , and geology[2].

The success of a state-of-the-art APT facility relies, not only on the procurement of an APT instrument but also on expert support in sample preparation, data analysis, and complementary electron microscopy. Hultgren Laboratory is well suited in this perspective considering prior engagement in APT research, with already well-established sample preparation and data analysis capabilities for APT as well as electron microscopy etc. in the laboratory.

[1] G. Sundell et al., Small. Vol. 15 (24).
[2] DW Saxey et al., Scripta Materialia 148, 115-121.

APT Instrument Specifications and Applications

Technical description

The proposed APT instrument will have the specification of an AMETEK-CAMECA LEAP 5000 XR, see Figure 2, or better. It is equipped with both voltage-assisted and laser-assisted evaporation of ions. The technical specifications are listed in Table 1.

Table 1*. CAMECA LEAP 5000 XR specifications.*

- ✓ Max. Voltage pulse frequency: 250 kHz.
- ✓ Mass resolving power (MRP): 1100 FWHM.
- ✓ Max voltage pulse fraction: < 25 %.
- ✓ Laser pulsing Frequency: 50 to 500 kHz.
- ✓ Wavelength: 355 nm.
- ✓ Laser pulse energy: 0.001 pJ – 1000 pJ.
- ✓ Laser spot size: < 3 µm.
- ✓ Specimen temperature: 20 – 100 K.
- ✓ Detection efficiency: ~52 %.
- ✓ Chemical detection limit: ~ 5 ppm.
- ✓ Spatial depth resolution: < 0.1 nm.

Figure 2. *Cameca LEAP 5000*

Instrument evolution

During the last decade, significant improvements to the LEAP system have been made to improve the ease of use, throughput, application areas, and data quality of the measurements.

Table 2. *Key improvements LEAP 3000 launched in 2007, LEAP 4000 launched in 2010, LEAP 5000 launched in 2015.*

	LEAP 3000	LEAP 4000	LEAP 5000
Detection efficiency	37 %	37 %	52 %
Data acquisition [Nr. of ions/hr.]	2 000 k	~2 500 k	4 000 k
Mass resolution (FWHM)	1000	1000	1100

The LEAP 3000 is available at Chalmers University and it was made by a prior manufacturer Imago Scientific, acquired by now sole developer AMETEK-CAMECA in 2010. The main 3^{rd} versus 5^{th} generation developments will improve the accuracy of the analysis volume by ~40 %, +2x data acquisition speed, and increased mass resolution by 10 %. The minimum time to measure one sample in the LEAP 5000 instrument is ~1.5 hrs. Although, sample preparation is the throughput limiting factor, nowadays, industry researchers have the expertise to do their own sample preparation but from KTH's side capacity has been upscaled as well.

Thus, since the introduction of the LEAP system APT has become a standard characterization method for the ultimate 3D nanoscale compositional characterization of metals, semiconductors, biomaterials, geological materials, etc. some of these applications are shown in Figure 3.

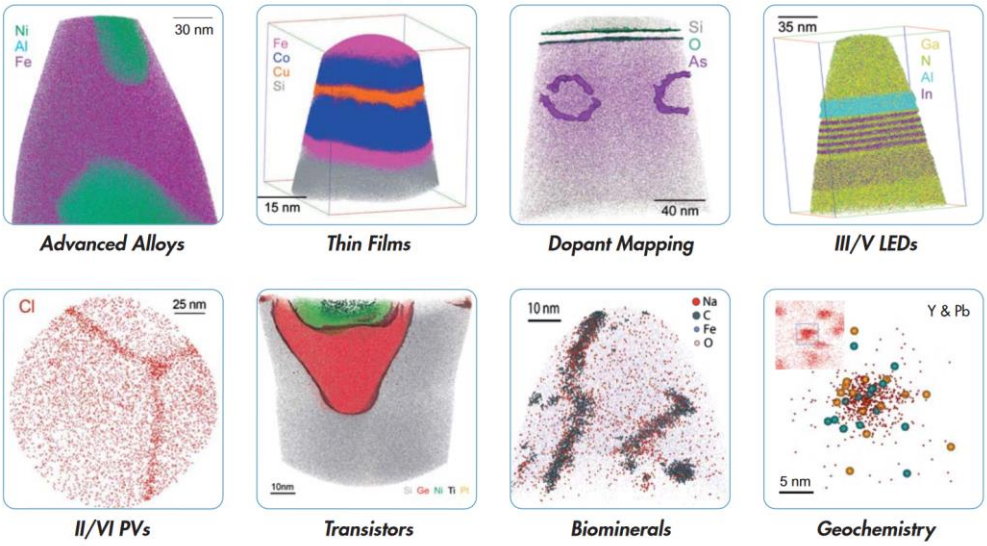

Figure 3*. Application of the LEAP 5000 X/R instruments (5th generation).*

The LEAP 5000 XR adds laser mode capability and extends the advantages in quantitative performance to the full range of LEAP applications. In addition, to the improved detection sensitivity, data quality, and specimen throughput, the advanced laser pulsing module enables increased yield with complex, fragile materials to enable new applications and make the LEAP 5000 XR the most versatile atom probe ever produced.

3 Way forward

We at the Hultgren laboratory seek your support in our applications for funding, firstly through the VR infrastructure grant. We would therefore like to invite you to an information meeting to present and discuss the project. We hope that you could provide us with useful input by sharing your potential scientific challenges, application areas, and known requirements on such an instrument.

We have scheduled two meetings for this purpose,

Friday, January 29th at 9.00 am.

Join Zoom Meeting
https://kth-se.zoom.us/j/61948887614

Wednesday, February 3rd at 9.00 am.

Join Zoom Meeting
https://kth-se.zoom.us/j/62242724739

R.S.V.P. to adahlstr@kth.se with name, affiliation, research field, and participation date to confirm your attendance.