Institution: University of Oxford

Unit of Assessment: UOA13

Title of case study: Developing the three dimensional atom probe

1. Summary of the impact

Research in the UAO has led to major advances in the technique of Atom Probe microanalysis. The UOA pioneered the concept of position sensitive detectors for Atom Probe instruments, generated the first 3D data and built the first prototype instruments. Following a series of patented advances and the formation of a spin-off company (subsequently incorporated into Ametek), research in the UOA has led directly to the sale of 45 Local Electrode Atom Probe (LEAP) instruments since 2008 with a value of $102M. These instruments have been used to provide atomic scale chemical information vital to the design of new commercial alloys and to safety cases for life extension of nuclear power plants.

2. Underpinning research

The 3-dimensional Atom Probe (3DAP) technique allows the study of the chemistry of engineering materials by evaporating a sample atom by atom and then reconstructing the local chemistry with sub-nanometer resolution into a 3D atomic scale image. The technique gives unique information on the phase chemistry and microstructure of engineering materials. Under the leadership of Smith and Cerezo in the UOA, two research themes have been pursued since 1998:

- advances in the performance and capability of the Atom Probe technique; and
- advances in metallurgy and engineering materials based on the application of the technique to a wide range of engineering problems.

Improvements in performance and capability resulting from research in the UAO included the idea of incorporating a reflectron lens to improve mass resolution by energy compensation of the extracted ions (European Patent No. 0231247, International patent PCT/ GB98/02678 16/09/98; US patent 6,580,069 17/06/03). This innovation enabled the complete separation of closely spaced mass peaks in spectra from complex alloys, and allowed the precise analysis of engineering materials not possible in earlier instrument designs [3.1]. Later, the UOA developed a movable extraction electrode and the 3-wire delay-line detector to replace wedge and strip anodes [3.2]. (UK patent 0003261.5, 15/08/01, EU 01102954.3, 16/0801, U.S. patent 6,661,013, 9/12/03). These two innovations improved the throughput of samples and increased the data collection rate by a factor of more than 100. A final stage of development was the wide-angle reflectron (U.S. patent 6,661,013, 09/12/03) that improved energy resolution in instruments incorporating moving electrodes in the voltage pulsing mode.

The UOA also exploited these developments in the study of a wide range of materials, generating more than 250 peer-reviewed papers. Key achievements included the first 3DAP studies of titanium and uranium alloys, investigation of precipitation reactions in commercial aluminium alloys and atomic scale chemical studies of semiconductor and catalyst nanostructures. A substantial body of work concerned precipitation reactions in nuclear pressure vessel steels [3.3]; for instance, the UOA published influential results on the nanoscale precipitation that embrittles steels, contributing to safety cases for the Sizewell B reactor. These publications included co-authors from more than 50 HEIs and more than 30 international industrial partners. The data are complementary, and of a much higher chemical resolution and sensitivity, than those available from electron microscopy, and publications from the UOA helped to establish the 3DAP as one of the most important tools for understanding the structure and properties of modern engineering materials [3.4].

A spin-out company was formed to develop the first commercial versions of the 3DAP, with Smith, Cerezo, Grovenor and Godfrey from the UOA as directors. In 1998 the company received *Millennium Product* designation for the Energy Compensated Position Sensitive Atom Probe, and this instrument was the forerunner of all commercial Atom Probe Instruments.

Based on the strong IP position developed in the UOA, the company was sold first to Polaron in 2002 and then in 2006 to Imago, now Ametek. Research and development in the UOA has thus led directly to designs critical for the performance of the current market-leading instrument, the Ametek-CAMECA Local Electrode Atom Probe (LEAP).

3. References to the research

The three asterisked outputs best indicate the quality of the underpinning research

The first report of a reflectron in a 3D Atom Probe

*3.1 *Performance of an energy-compensated 3-dimensional atom probe*. A. Cerezo, T.J. Godfrey, S.J. Sijbrandij, P.J. Warren and G.D.W. Smith, *Rev. Sci. Instrum.*, **69** (1998) 49-58. doi: 10.1063/1.1148477

First report of improved 3D detector performance

*3.2 *Multiple hit readout of a microchannel plate detector with a three-layer delay-line anode*. O. Jagutzki, A. Cerezo, A. Czasch, *et al.*, *IEEE Trans. Nuclear Science*, **49** (2002) 2477-2483. doi: 10.1109/TNS.2002.803889

Development of 3D Atom Probe techniques for nuclear materials

*3.3 *A procedure for quantification of precipitate microstructures from three-dimensional atom probe data* D.Vaumousse, A. Cerezo, and P.J. Warren, *Ultramicroscopy*, **95** (2003) 215-221 doi: 10.1016/S0304-3991(02)00319-4

3D Atom Probe analysis supporting development of a commercial material

3.4 *Atomic-scale structure of sputtered metal multilayers.* X.W. Zhou, H.N.G. Hadley, R.A. Johnson, D.L. Larson, N. Tabat, A. Cerezo, A.K. Petford-Long, G.D.W. Smith, P. H. Clifton, R. L. Martens and T. F. Kelly, *Acta Materialia*, **49** (2001) 4005-4015. doi: 10.1016/S1359-6454(01)00287-7

4. Details of the impact

The impact of the research in the REF2014 period of assessment is of three types:

(i) The generation of the market for Local Electrode Atom Probes

Over the period 2008 - July 2013, 45 LEAP systems have been installed in laboratories in Japan, USA, Germany, Korea and 12 other countries, representing sales of $102M, as can be confirmed by the Division Vice President of AMETEK, Inc.

(ii) The benefits arising from using the LEAP to generate of new scientific understanding to underpin safety cases for the nuclear industry

Atom Probe microanalysis has become a vital tool in the study of the changes in microstructure in reactor pressure vessel steels during prolonged irradiation. The LEAP makes a critical contribution to safety cases for life extension of GEN3 nuclear power plant, as demonstrated by the following testimonials:

(a) Senior Research Scientist: Central Research Institute of the Electrical Power Industry, Japan

"We have 3 LEAPs. The 2nd installed in 2008 and the 3rd in 2009 in our hot lab. One of the very

important applications of the LEAP in our institute is to study the effect of neutron irradiation on the microstructural changes in the reactor pressure vessel steels of the Japanese nuclear power plants. Currently we routinely perform LEAP analysis on surveillance materials from the RPVs of Japanese NPPs. The information obtained from this activity is very important to ensure the integrity of the RPVs. We mainly work for the Japanese electric utilities, and the benefit they obtain through the LEAP analyses of their materials is huge."

(b) Professor in Materials Department: University of California, Santa Barbara, USA

*"We purchased our LEAP in January 2009 and we use it as a central part of our research on an advanced, potentially technologically trans-formative, nano-structured ferritic alloy for fission and fusion applications with remarkable high temperature properties and irradiation tolerance. In this project the LEAP is not just important but critical. We are also using the LEAP as **THE** core tool in studying radiation damage to materials used in light water reactors to provide a basis for nuclear plant life extension. Without the LEAP it would be difficult, if not impossible, to develop data and models to predict the behavior of materials in reactors over periods up to 80 years or more. Nuclear power is the largest source of C-free electricity in the US and the economic impact of extended life is enormous - ultimately totaling at least in the many tens of billions of dollars range."*

(iii) **The benefits resulting from the use of the LEAP in the development of new commercial alloys**

The impact of the LEAP in alloy development is demonstrated by the following testimonials from senior staff in leading international laboratories:

(a) Director and Head of Department: Max-Planck-Institut für Eisenforschung GmbH

"The Max Planck Society is Germany's most successful research organization. The Research Group for Atom Probe Tomography at the Max-Planck-Institut für Eisenforschung was established in 2010 with the installation of a LEAP. It is not just important but it is an absolute breakthrough technology for materials engineering, it is essential for us, and the research by the University of Oxford has been at the absolute forefront here: the international community truly owes a lot to Oxford! Examples for applied projects which we can ONLY pursue through APT include:

1. *Development of new Mn - based maraging steels.*
2. *Development of new pearlite wires as used in type cord, bridges etc.*
3. *Development of new thin film solar cell materials.*
4. *Development of new hard metal-nitride multilayer coatings for drilling applications.*
5. *Development of new superalloys for 700°C coal power plants."*

(b) Professor, Chalmers University of Technology

"We purchased a LEAP in 2008, and it is such an important part of our materials analysis capability that we established a Materials Analysis Laboratory as a separate unit studying steels, superalloys, zirconium alloys and hard materials. One major project is a new steel for steam power plants. The thermal efficiency is limited by the maximum service temperature of the steel used for components such as turbine rotors and casings. Today the maximum temperature is 600°C; limiting the efficiency to about 45% in the most modern plants. We aim to increase the service temperature to 650°C by a new alloying concept. This would give 50% efficiency and enormous savings in fuel consumption and carbon dioxide emissions world-wide. This development work relies heavily on the ability of LEAP to analyse very small precipitates. The work is done in collaboration with Siemens, Saarschmiede and RWE, ensuring swift scaling up to commercial products."

5. Sources to corroborate the impact

New Market: The sales figures quoted above, and the impact of research in the UOA on the development of the LEAP, can be corroborated by the Division Vice President of AMETEK, Inc

The quotations above on the impact of the LEAP to the nuclear industry can be corroborated by:

Senior Research Scientist: Central Research Institute of the Electrical Power, Japan

Professor: University of California; Santa Barbara, USA

The quotations above on the impact of the LEAP to alloy development research can be corroborated by:

Director and Head of Department: Max-Planck-Institut für Eisenforschung GmbH, Germany

Professor: Chalmers University of Technology, Sweden