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► To cite this version:

A. Cerezo, T. Godfrey, A. Moore, G. Smith. AN ELECTRONIC AND COMPUTER SYSTEM FOR AN AUTOMATED ATOM PROBE. Journal de Physique Colloques, 1984, 45 (C9), pp.C9-315-C9-321. 10.1051/jphyscol:1984953 . jpa-00224439

HAL Id: jpa-00224439 https://hal.science/jpa-00224439

Submitted on 4 Feb 2008

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AN ELECTRONIC AND COMPUTER SYSTEM FOR AN AUTOMATED ATOM PROBE

A. Cerezo, T.J. Godfrey, A.J.W. Moore and G.D.W. Smith

Department of Metallurgy and Science of Materials, University of Oxford, Parks Road, Oxford OX1 3PH, U.K.

<u>Résumé</u> - Un système électronique qui contrôle les tensions, mesure les temps, <u>calcule les masses et affiche les spectres a été appliqué à la sonde à atomes</u> VG Scientific FIM 100. Le taux de désorption d'ions est contrôlé automatiquement par ajustement de tension lorsque le courant ionique s'écarte de limites consignées. Les fonctions simples de l'instrument, telles que l'IAP peuvent être accessibles sans passer par le calculateur et le réglage de tension manuel est toujours possible. Les résultats sont stockés sur un disques et peuvent être analysés après coup en utilisant le même programme. D'autres systèmes en temps réel donnent les changements de composition avec la profondeur, le rapport m/e d'ions successifs, des diagrammes "échelle", ou la comparaison de deux spectres où les deux peuvent être complétés ou bien un mis en mémoire. Les séquences peuvent être imprimées et les spectres et autres diagrammes tracés. Le taux de désorption élevé combiné à l'analyse en temps réel permettent d'avoir un système efficace.

<u>Abstract</u> - An electronic system which controls voltages, measures times, calculates masses and displays spectra has been applied to the VG Scientific FIM100 atom probe. The rate of ion desorption is automatically controlled by voltage adjustments which occur when the ion rate falls outside preset limits. Simple instrument functions, for example, use as an IAP, can be carried out with or without computer control, and manual voltage control is available at all times. The experimental data are stored on a disc and can be output later and re-analysed using the same program. Other real time screen displays include plots of changes of composition with depth, sequences such as the mass to charge ratio of successive ions or related quantities, "ladder diagrams", or the comparison of two spectra where both may be continually updated or one may be a stored spectra taken at an earlier stage of the experiment. Sequences can be printed out and the spectra and other diagrams can be plotted. The high rate of ion desorption combined with analysis in real time provides a very efficient system.

This paper describes the electronic system for an automated atom probe fitted with a Poschenrieder analyser [1], and imaging atom probe (IAP) and having facilities for both voltage and laser pulsing of the specimen. It has been applied to the VG Scientific FIM100 atom probe which was delivered to Oxford early in January 1984 [2]. The two parts of this paper will describe (1) details of the electronic equipment (2) the computer control of the system.

1 - THE ELECTRONIC EQUIPMENT

The design of the electronic system has followed two main principles:

(i) The system is to be automated whilst also allowing manual control, the use of the instrument as a FIM and IAP without a computer and giving protection C9-316

to the specimen in the case of a power failure.

(ii) All interfaces within the system must be standard, allowing greatest flexibility in accommodating modifications, e.g. use of a different computer.

Thus, the system is to some degree modular (Fig.1), the heart of this being the acquisition and control units for the atom probe which are all CAMAC (IEEE-583) modules. These are controlled by the computer via a CAMAC-to-GPIB (IEEE-488) interface whilst they in turn control the high-voltage power supplies and read the times-of-flight of desorbed species. The GPIB also allows linking of the computer to a transient digitiser, where spectra from the IAP may be recorded, and to the necessary printer and plotter for hardcopy output.



Fig.1 Schematic of the electronics system.

Fulses from the channel-plate detector, after amplification, shaping and discrimination, are fed to a LeCroy 4208 Time-to-Digital Converter [3]. This measures times of up to 8 milliseconds in each of 8 channels with a resolution of 1 nanosecond. A simple logic module is used to gate the input to the timer and clear it on cycles where no ions are caught, leaving the computer free for other tasks. On detecting an ion within the given time window, pulsing is stopped and a service request issued to the computer. Once the computer has read the times-of-flight and number of unsuccessful pulses it may then restart pulsing. A 16-channel, 12-bit digital-to-analogue converter allows computer control of DC, pulse and Poschenrieder voltage supplies whilst an analogue-to-digital converter measures these voltages and checks for any malfunction of the automatically controlled power supplies.

A small remote unit allows easy control of the specimen voltages, mainly during use of the instrument as a FIM. In automatic mode this is switched out but maintains the voltage given by the computer. Thus, with any power failure, the system returns to manual control and the specimen is protected.

When the IAP is in use, the transient waveform recorder (Tektronix 7912 AD) is fed from the double curved channel-plate used for the detector and screen assembly. In real time the recorder works as an oscilloscope or storage oscilloscope and allows accurate setting of the time-gating pulses required for selective imaging of desorbed species. Under control of the computer, the waveform can be digitised and stored on disc; the spectra thus obtained provide a semiquantitative analysis from a large part of the imaged area of the specimen.

2 - AUTOMATIC OPERATION OF THE SYSTEM

The program was planned so that the operator can control the equipment from a keyboard and display the results on a screen. The raw experimental data (flight times, voltages, number of unsuccessful pulses between detected ions) can be stored on disc so that if required, the stored data can be extracted later and reanalysed by the program. A printer is available to keep a timed record of messages from the operator, or of voltage changes or to enable the print out of any of the ion Similarly a plotter provides copies of any mass spectrum or other sequences. displays selected by the operator. Finally there are messages on the screen which remind the operator about the options available at that time. The system was built around a Sirius computer with 512K RAM, 2 floppy disc drives, 15Mbyte hard disc, Epson FX80 printer, and an Itoh CX4800 drum plotter with buffer. The main considerations for selecting this system were screen resolution, flexibility of keyboard configuration, IEEE-488 interfacing, and a large memory. The source code is in Pascal and is written to be easily transferable to other machines.

(a) Operating Modes. The system will operate in several modes.

- (i) The desorption pulses may be either laser or voltage pulses.
- (ii) The instrument operates with a probe hole or as an imaging atom probe.

(iii) The raw data (time of flight etc) may either arrive in real time during the experiment or may be derived from a disc file made in a prior experiment.

At power up a screen display informs the operator of the current modes and provides instructions for alteration of the modes from the keyboard. If necessary the modes are changed and then the next display is obtained via carriage return.

(b) Runtime Constants.

The next display are of the values of several constants which affect the course of the experiment (Figs.2 and 3). In Fig.2 Nos 1-3 determine the conditions for the termination of the experiment. Nos. 4,5 determine how the voltage is controlled. Nos. 6-10 affect the conditions for voltage adjustment and the amount of adjustment. Nos. 11-15 set up file names and voltages at the start of the experiment. No. 16 produces a new screen display of further constants which are changed less frequently. In Fig.3, Nos. 5-8 are constants in the equation relating mass/charge to the time of flight and Nos. 9,10 are the voltage ratios for the Poschenrieder analyser. The operator can change the value of any constants and then the next display is obtained via carriage return.

RUNT IME CONSTAN	<u>ITS</u>
1 MAXIMUM DC VOLTS FOR EXPERIMENT. 2 Maximum Pulse Volts For Experiment. 3 Maximum Andrea Dr Lons. 4 Comtrol of DC or Pulse Voltage. 5 Automatic or Manhum Voltage Adjustment 6 Ion Quota for Voltage Adjustment. 7 Upper Linit of Mull Pulses In Quota. 8 Voltage Internation. 18 Voltage Decrement. 19 Unare Linit of Mull Pulses In Quota. 19 Unare Linit of Mull Pulses In Quota. 19 Starting Pulse Voltage. 12 Starting Pulse Voltage. 13 Message to End Data File. ((70 Chara	((38888) 18688 ((78688) 5688 ((386880) 15688 ((0 or P) PU ((1 or P) PU ((21) 5 ((3999) 28 ((3999) 28 ((3999) 2 ((3999) 2 ((3999) 2 ((3999) 2 ((3999) 2 ((3999) 2 ((3999) 15 ((3999) 16 etcters. END with CR). 8
14 MARE OF THE INITIAL DATA FILE.(<9 CHAR 15 MARE OF THE RAN DATA FILE.(<9 CHARS)++ 16 CHARGE TO MIXILIARY SET OF RUNTIAE CO 17 RETURN TO MODE SELECTION. To change constant-Type the selected mo(*CR) Type CR for numerical dis	IS)(+CR) INITDATA +CR) TUNGSTEM INSTANTS),then the new value(+CR). splay.

Fig.2 - The display of runtime constants showing the default values which can be modified by the operator.

AUXILLIARY RUNTIME CONSTANTS		
1: HAXIMUH HULL PULSES ((*9999) 2: INCLUBE TOFS ON DISC FILE (Y or N) 3: INCLUBE TOFS ON DISC FILE (Y or N) 3: INCLUBE TOFS ON DISC FILE (Y or N) 4: PULSE DELMY (MAND SEC) ((*9999) 5: LOUER LINIT OF TOF (MAND SEC) (*99) 6: ALPMA-ULTAGE FACTOR (<2.0) 7: BETA-PULSE VULTAGE FACTOR (<2.0) 9: OFFLETOR I (ULTAGE FACTOR (VOSIT)UE FACTION). 10: DEFLECTOR I (ULTAGE FACTOR (VOSIT)UE FACTION). 10: BEFLECTOR I (ULTAGE FACTOR (VOSIT)UE FACTION). 11: INSTRUMENTAL NAX UULTAGE FOR DC (<22080) 12: INSTRUMENTAL NAX UULTAGE FOR PLUSE (<228080) 13: FLIGHT TUBE LENGTH (•m) (ILFGE FOR RUTINE CONSTRUTS 14 OHMEG TO RNICTINE SET OF RUNTINE CONSTRUTS 15 RETURN TO NODE SELECTION	1000 Y P 18 1.000 1.259 0 0.2220 0.1940 30000 7000 2265	
To change constant-Type the selected no(+CR), then the new value(+CR). Type CR for numerical display.		

Fig.3 - Further runtime constants which will be changed infrequently.

(c) Numerical Display

This display shows many of the parameters which are changing during the course of the experiment (Fig.4). The display shows the background and pulse volts, the number of ions detected, the number of unsuccessful pulses and several other totals which will be described later.

UOLTS PROGRAMMED PULSES BACKGROUND UOLTS 9948 PULSE UOLTS 13489 TOTAL IONS 2176 NULLS IN QUITA 57 NULLS SINCE IOM 18 SAMPLEC 1880 95 T IN SAMPLE 93 B IN SAMPLE 8	TIME CHANNELS(NS) 12807 12802	WINDOWS 1 66-5-62.2 2851 2 45.4-46.6 188 3 8.8-8.8 4 8.8-8.8 5 8.8-8.8 6 8.8-8.8 6 8.8-8.8 8 8.9-8.8 8 8.9-8.8 8 8.9-8.8 8 8.9-8.8 8 8.9-8.8 8 8.9-8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8		
C IN SAMPLE 0 CONCENTRATION ORDER 0 LADDER SPECIES 08 SPECTRUM 01 50.0- 64.0 LIM U 02 30.0- 90.0 LIM 1 SPECTRUM 01 0.1-250.0 LIM 0 82 0.1-250.0 LIM	CURSOR M/H 60.7 CURSOR IONS 367 1 AREA COUNT 603 1 1 14 T 1 100000 15 B 8 8 8 8 8 8 8	8 8.8- 8.8 8.8 9 8.8- 8.8 8.8 10 8.8- 8.8 8 11 0.8- 8.8 8 12 8.8- 8.8 8 13 8.8- 8.8 8 8 8 8 8 8		
SPECTRUM C1 0.1-250.0 LIN 0 C2 0.1-250.0 LIN	IĞĊ ÖÖÖÖÖÖ	8888888 8888888 8888888888888888888888		
SEQUENCES: Type I for ions/pulse. It for masses. S for species -repeat to cancel. Type CR for Runtime Constants or use Special Keys.				

Fig.4 - The numeric display which shows changing totals while ions are being collected. The windows 1 and 2 are set for tungsten with 3 and 4 charges and window 13 is set for all tungsten ions. The flight times of the last ions detected are shown in the middle of the screen.

(d) Programmable Keys and Pulsing

The operation is controlled from the keyboard with the 10 programmable keys. They have different functions in normal and in shift mode and are separately labelled. Pulsing is started with the CONTINUOUS PULSING or SINGLE PULSE keys. Whenever ions are detected their flight times are briefly displayed in the middle of the numerical display and the appropriate totals are updated.

(e) Spectra

The SPECTRUM DISPLAY key provides a spectral display of the mass to charge ratios of the ions which is updated in real time (Fig.5). The horizontal scale has a maximum range of 0-250 amu and may be changed to any intermediate range. All masses are calculated to 0.1 amu and the screen resolution is 600 spectral lines. The vertical scale can be linear or logarithmic and will automatically adjust if any line reaches the maximum value. The top two lines of the screen are a summary of the information from the numerical display and are available on all other graphic displays. Two spectra may be displayed simultaneously (Fig.6) and they may be different parts of the same spectrum, or one spectrum may be a detail of the other, or only one of the spectra may be updated while the other is a fixed display selected earlier in the experiment. The program stores details such as the horizontal and vertical limits of 6 spectra. The spectra are termed Al,A2,Bl,B2,Cl,C2 and initially all have horizontal limits of 0-250 amu. The A spectra are updated in real time and the B and C spectra are used to store the current spectrum at any time by using the RESERVE SPECTRUM key. The digits 1 and 2 provide the capacity to set different limits for the same spectra. The operator can thereby switch spectra without resetting the limits. The numerical display shows the current settings of the limits for the 6 spectra. A cursor is available, operated by the arrow keys and can be used to identify and measure lines, count ions between two selected masses or to select window limits.





Fig.5 - A spectral display of tungsten ions (3+) which is updated as ions are detected.

Fig.6 - Two spectral displays of tungsten ions with the upper spectrum being a detail of the lower spectrum.

(f) Mass Windows

The SET WINDOWS key produces instructions for selecting the mass limits for up to 13 windows in the spectrum so that ions of a particular species may be identified and counted. Successive key strokes provide the number of the window and then the upper and lower mass limits. Different windows may be used to identify a species in different charge states. Three additional windows numbered 14-16 are termed dependent windows and can be defined by 13 coefficients which are multiplied by the totals in windows 1-13 to give the total ions in one species or in some combination of species. The coefficients may be zero or any small integer. The dependent windows may be allotted a window identifier character which represents the species (the default characters are A,B and C). During pulsing the window totals in the numerical display are updated.

(g) Control of Ion Rate

During pulsing, the number of ions per pulse fluctuates due to the passage of the plane edges over the probe hole and also the ion rate decreases due to a continual increase in the specimen radius. The program adjusts the voltage to keep the ion rate within limits specified by the operator. When the number of unsuccessful pulses occurring during the detection of a selected number of ions (ion quota, Fig.2) exceeds a given maximum (upper limit of null pulses, Fig.2), the voltage is increased by a chosen amount (voltage increment, Fig.2). Similarly if the ion rate becomes too great the voltage is decreased (lower limit of null pulses, voltage decrement, Fig.2). When the voltages changes the new value can be output on the printer.

(h) Depth Profiles

The PROFILE DISPLAY key provides the means of measuring the number of ions of different species which occur in successive samples of ions. The size of the sample and the species to be counted may be selected by following instructions on the screen. Up to three species may be used which are identified by their window identification character. The screen display then shows a plot of the concentration of the selected species in successive samples. The totals of each species in the current sample are shown in the numerical display.

(i) Sequences

Four sequences are available which are useful in observing the course of the experiment.

(i) A sequence of the number of unsuccessful pulses and the number of ions detected by each successful pulse.

(ii) A sequence of the masses of the ions detected.

(iii) A sequence of the species (shown by the window identification character) and other ions (shown as .).

(iv) A sequence of the flight times of the detected ions.

The numerical display provides instructions for displaying one or more of the sequences (i), (ii) and (iii) in real time. The area of screen available restricts the display to one wrap around line for each sequence.

Longer sequences are obtained with the DISPLAY SEQUENCES key which gives instructions for a choice of any of the four sequences. The display of the selected sequence uses the whole screen with scrolling. The PRINTING key can be used to select and output any sequence to the printer.

(j) Ladder Diagram

A ladder diagram [4] is a graphical method of detecting changes in the relative numbers of two species (a and b). Each time a or b is detected the plot moves one unit in the horizontal or vertical direction respectively. Changes in slope which indicate changes in relative concentration can be detected easily. The LADDER DISPLAY key provides instructions for selecting the two species and their ladder diagram is then plotted in real time. The species in the current ladder display are shown in the numerical display.

(k) The Plotter

Use of the PLOTTING key gives instructions for choosing one of the six spectrum plots, a ladder diagram or a depth profile which is then plotted.

(1) Messages to the Printer

When the ECHO key is used the current time and number of ions detected is printed and a message may now be typed to the printer. The printout can therefore be a complete record of ions detected, times, messages, voltage changes and any sequences requested by the operator.

(m) Storage of Raw Data

In the display of runtime constants (Fig 2) numbers 15 and 13 provide the opportunity to type in the name of the raw data file and a descriptive heading. The file is generated on the hard disc during pulsing and contains flight times, voltages and the numbers of unsuccessful pulses. At the end of a useful experiment it is possible to transfer the file to a floppy disc for future analysis with the same program.

3 - CONCLUSIONS

It is acknowledged that the development of the system has drawn on some features incorporated in other atom probe systems. The result is a large variety of coordinated facilities. An important feature is that several of the facilities may be available simultaneously. There could be a spectrum displayed on the screen, a sequence being output on the printer and a ladder diagram on the plotter, all of them recording ions currently being detected. The displays can be immediately changed by using the programmable keys and following the instructions on the screen. The operator is always informed of the current options.

The high rate of ion collection combined with the immediate analysis of results produces a highly efficient system. The preparation chamber of the VG FIM100 makes it possible to introduce, prepare and store specimens in the preparation chamber during ion collection, and delays between experiments are thereby reduced. The desorption probe and the laser are now being installed and automated. It will then be possible to analyse the specimen by all these techniques and to resharpen it by ion thinning whenever it becomes too blunt, without removing it from the system.

Acknowledgements

Financial support for this project was provided by the Paul Instrument Fund of the Royal Society, the Science and Engineering Research Council (SERC), and the E.P.Abrahams Trust. AJWM wishes to thank the Royal Society for the award of an Anglo Australasian Visiting Fellowship during 1982-83 and SERC for the award of a Visiting Fellowship during 1983-84. AC wishes to thank VG Scientific for the provision of a CASE studentship. We also thank Professor J.W.Christian, FRS, for the provision of laboratory facilities.

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