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Remote atom probe field ion microscopy

M.K. Miller*, D.J. Larson

Microscopy and Microanalytical Sciences Group, Metals and Ceramics Division, Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6376, USA

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Abstract

Each of the normal stages of operation of the atom probe, i.e., specimen loading, field ion imaging, selection of the feature of interest for analysis, and atom probe analysis, have been examined to determine the feasibility of operating an atom probe over the Internet. Experiments have been performed that have demonstrated the feasibility of remotely monitoring and controlling a conventional atom probe field ion microscope and a three-dimensional atom probe. Published by Elsevier Science B.V.

Keywords: Atom probe analysis; Remote monitoring

1. Introduction

In the current economic climate, there is a growing requirement to reduce the cost of research, increase the usage of specialized microscopes, and make these instruments available to a larger number of researchers. One potential solution to this problem is to increase the availability of these instruments in such a way that they may be controlled remotely over the Internet. The specialized nature and low number of high-quality atom probe field ion microscopes and three-dimensional atom probes (3DAP) around the world makes them prime candidates for this type of operation.

There are several levels of operation that could be considered ranging from simple remote monitoring of the instrument, complete control by a remote user, to fully automatic operation including sample loading. The fully automatic mode of operation also has some major advantages for the examination of highly activated neutron-irradiated materials. Each of these approaches would require some modifications to the standard instrument and the method by which it is controlled.

In this paper, each of the normal stages of operation of the atom probe, i.e., specimen loading, field ion imaging, selection of the feature of interest for analysis, and atom probe analysis, have been examined to determine the feasibility of operating an atom probe field ion microscope remotely over the Internet.

In addition, some experiments have been performed to evaluate the workability of remotely monitoring and controlling a conventional atom probe field ion microscope and a three-dimensional atom probe.

2. Typical atom probe experiment

In order to evaluate what is required to adapt a typical atom probe field ion microscope [1] into

* Corresponding author. Tel.: + 1-423-574-4719; fax: + 1-423-574-0641.

E-mail address: xkm@ornl.gov (M.K. Miller)

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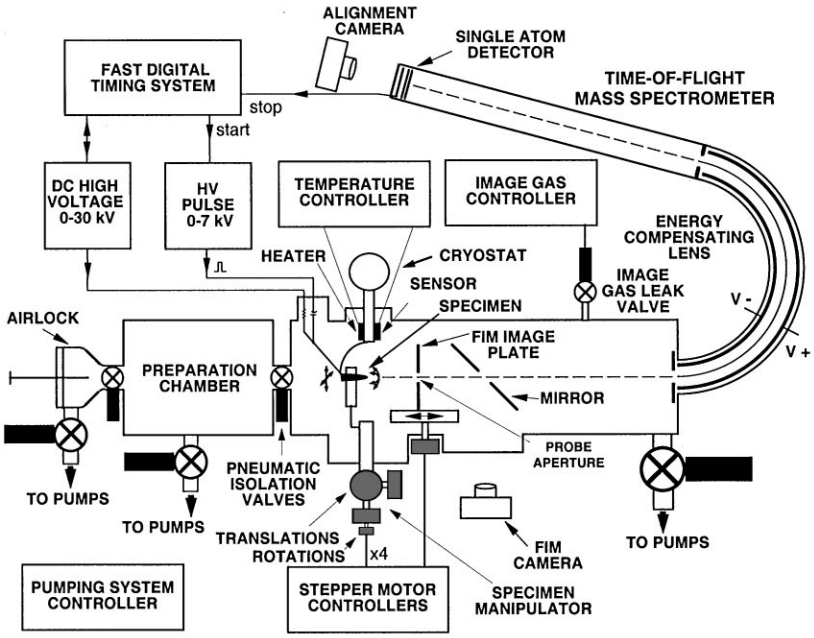

Fig. 1. Schematic diagram of a conventional atom probe field ion microscope configured for remote operation. The fast digital timing system, temperature, image gas, pumping system, and stepper motor controllers are interfaced to a local computer.

an instrument that may be remotely operated, consider a typical atom probe experiment that consists of the following sequence of events:

(a) fabricate the field ion specimen,

(b) load the specimen into the atom probe,

(c) pump the system to a base pressure of less than 2×10^{-8} Pa,

(d) cool specimen to a cryogenic temperature (typically 15–70 K),

(e) admit an image gas to a pressure of $\sim 2\text{–}5 \times 10^{-3}$ Pa,

(f) activate the imaging microchannel plate and phosphor screen,

(g) slowly apply a positive high voltage to the specimen to form the field ion image and field evaporate the specimen to a desired point,

(h) rotate the specimen so that a feature is selected for analysis in the atom probe,

(i) activate single atom detector and energy-compensating lenses,

(j) align the specimen to the optical axis in a conventional atom probe by translating the specimen,

(k) remove the image gas from the system, (note: this operation is typically performed before (i) in a 3DAP), and

(l) apply high voltage pulses to the specimen and monitor the electronics associated with the time-of-flight mass spectrometer and single atom sensitive detector system.

At the completion of the experiment, events (l), (i), (g), (f), and (b) have to be turned off or performed in reverse.

A schematic diagram of an atom probe field ion microscope configured for remote operation is shown in Fig. 1. The features required for remote operation are described below.

Although field ion specimens have been successfully fabricated from bulk radioactive material completely within a remotely operated hot cell environment, it is impractical to automate the process due to the diversity of the materials to be examined and the delicate nature of the specimen.

Specimen loading is generally performed in three stages; loading into a fast entry airlock, transferring into a preparation chamber, and finally transferring

to the specimen stage in the analysis chamber. Although, it would be possible to perform these operations under robotic control, this method would not be cost effective as a local operator would still be required to initiate the process.

Since most modern commercial atom probes feature semiautomatic pumping systems, automation of these systems for stages (c), (e) and (k) under computer control would require only minor modifications. Ultrahigh vacuum compatible gas controllers are also commercially available to leak the required amount of image gas into the system. The temperature of the specimen (d) may be changed by a standard temperature controller.

Specimen manipulation [stages (h) and (j)] may be computer controlled by the simple addition of stepper motors to the two rotations and two translations on the specimen stage, as shown in Fig. 1. A stepper motor could also be incorporated into the imaging channel plate and phosphor screen assembly (or an equivalent iris mechanism) so that the effective size of the probe aperture may be automatically maintained during a conventional atom probe analysis.

The remaining processes are discussed in more detail in the following sections.

3. Remotely monitoring the experiment

The simplest method is to view the field ion image and the output from an atom probe analysis on a remote computer while a local operator controls the instrument. The use of video cameras to view the field ion image was first performed by Panitz [2]. This recording process may be computerized with the incorporation of a frame grabber or equivalent device in the control computer. Experiments have demonstrated that typical metallurgical applications require a high resolution [greater than 512×512], low light video camera. As a typical field ion image is made up of many thousands of small bright dots of varying intensity in a dark background, the standard image compression techniques used to compress the image before transmission over the Internet are relatively inefficient and therefore, relatively large amounts of data are required to be transferred from the control computer to the remote site. The frame refresh rate for a field ion image over the Internet has been measured and is typically in the tens of seconds at the present time.

This method of operation has been achieved over the Internet with commercially available software, as shown in Fig. 2. In this atom probe analysis of a commercial nickel-based superalloy, the progress of the experiment as the cylinder of analysis samples the γ, γ' and γ'' phases is monitored from the composition profile.

This monitoring arrangement permits the remote user to instruct the local operator which feature to select in the field ion image or which area to analyze and then monitor the progress of the atom probe experiment from composition profiles or atom maps, etc. Monitoring may also be used to terminate the experiment after an appropriate number of atoms have been collected or when the feature is no longer in the analysis volume.

4. Remotely controlling the experiment

Preliminary experiments have revealed that field ion microscopy and the field evaporation process do not easily lend themselves to full remote control. The primary reason for this difficulty during the field ion imaging portion of the experiment is that rapid feedback in the form of monitoring the field ion image and the rate of field evaporation of material is required to prevent applying an excessive voltage to the specimen and consequently destroying the specimen. Since the image refresh rate is low, safeguards are required to minimize this possibility.

A remotely controlled field ion microscopy experiment of a Ni_4Mo phase is shown in Fig. 3. The voltage applied to the specimen may be increased or decreased either by remotely pressing the ↑ and ↓ buttons or by directly entering the desired voltage. When the applied voltage is approximately at the best image voltage of the specimen, the voltage increments for the changes should be relatively small. The precise value of the step used for the voltage increase depends on the difference between the best image voltage and the onset of field evaporation. This parameter is material and

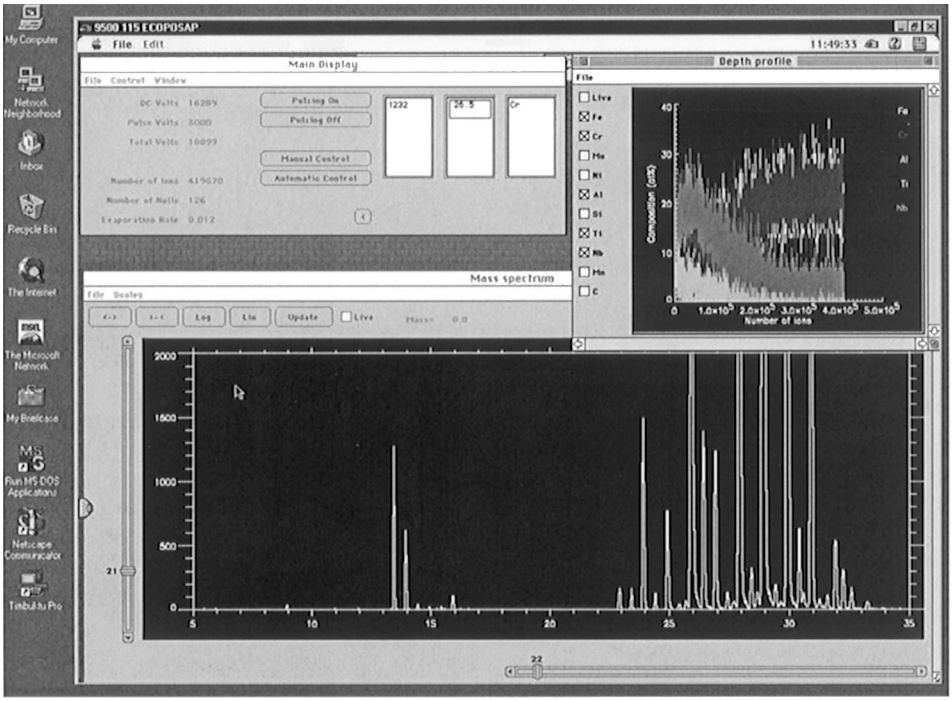

Fig. 2. Live screen dump during a 3DAP experiment of a nickel-based superalloy. The remote monitoring computer (full screen) displays the state of the experiment in the form of a mass spectrum, a composition or depth profile and status (inner screen). Note the different computer platforms used in this experiment (see title bars on inner screen).

best-image-voltage dependent. It was found to be more practical to have a larger default step for the decrement than for the increment and to be advantageous to be able to change these increments on-line.

Pulsed field evaporation may also be performed by pressing the "Pulsing On" and "Pulsing Off" buttons after selecting the desired pulse fraction. Experiments have revealed that pulsed field evaporation was simpler to perform than increasing the standing voltage in small steps.

Most modern computer-controlled atom probes are designed to automatically increase the standing and pulse voltages on the specimen and the corresponding voltages on the energy compensating lenses to maintain a constant ion collection rate, controlling the application of the field evaporation pulses to the specimen, and monitoring the flight times from the mass spectrometer. In addition, the control programs are designed to run unattended so that the instruments may be used continuously. A typical experiment may continue for up to 24 h in a 3DAP. Therefore, there is little distinction between local and remote operation after the data acquisition has started. It should be noted that a well-designed control program that features a graphical user interface can potentially be used for remote control without modification.

Portions of a prototype design of a fully computer-controlled atom probe are shown in Fig. 4. In this prototype design, all isolation and leak valves, vacuum gauges, specimen temperature controller, rotation and translation motions of the specimen stage are under the control or monitor of the computer. Although this program has not been fully implemented, the functionality of most of the critical subsystems (specimen temperature control and monitor, specimen rotation and translation, pressure monitoring and valve control functions) have been independently demonstrated on test systems.

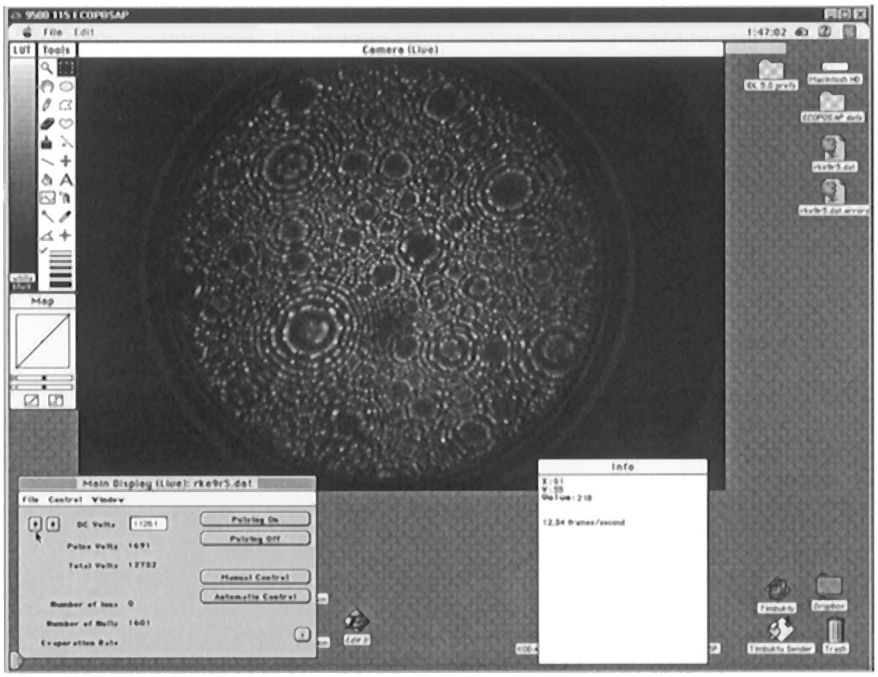

Fig. 3. Live screen dump during a field ion microscopy experiment of a Ni_4Mo phase. In this experiment the remote computer is controlling the experiment and all the buttons are active. The voltage applied to the specimen may be adjusted by the buttons (arrowed) or by entering the desired voltage (lower left). Pulsed field evaporation may also be performed by pressing the pulsing on and off buttons.

5. Automatic operation

In order to operate an atom probe in a fully automatic mode, the operations outlined in Section 2 could be fully automated. The main difficulties with an unattended mode of operation are to apply the correct voltage to the specimen to form a stable field ion image and then to select a desired feature for analysis. Unfortunately, monitoring the rate of image gas ions produced by the field does not yield a suitable signal to define the best image voltage as the ion current can both increase and decrease as the best image voltage is approached. In addition, the average or peak levels of the video signal on the camera are not good reference points for the field ion image. Therefore, the best image voltage would have to be estimated directly from the field ion image with some sophisticated artificial intelligence algorithms. However, these algorithms are not available at the present time and they would require a significant effort to implement the required database for a wide range of materials. In addition, most specimens require some field evaporation to develop a suitable end form before analysis. However, this imaging step could be eliminated and the specimen could be analyzed solely in the time-of-flight mass spectrometer. One minor problem with this approach is that the current microchannel plate-based single atom detectors have low detection efficiencies for low-energy ions (< 3 kV) and therefore extremely sharp specimens may not survive. In addition, the type of analysis that could be performed would be limited to random area analysis [1] unless sophisticated artificial intelligence or pattern recognition algorithms could be developed to correctly identify microstructural features in the field ion images.

6. Summary

This study has shown that a relatively small number of modifications are required to convert an

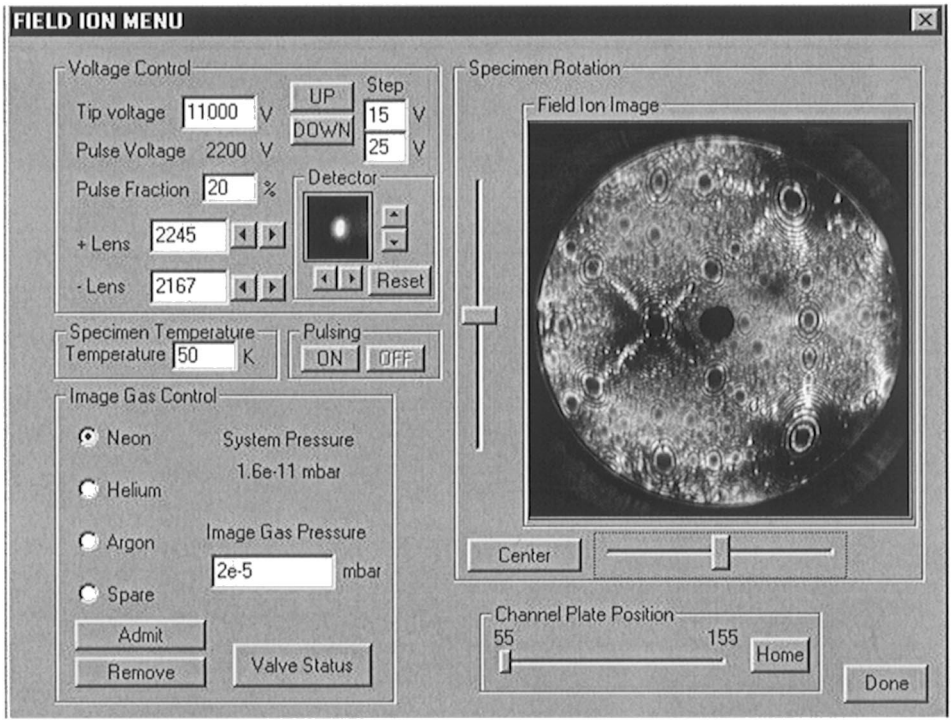

Fig. 4. A menu from a prototype program designed to remotely perform the alignment and specimen positioning of an energy-compensated atom probe. The sliders adjust the position of the specimen via stepper motors in the specimen stage and channel plate system and the translation of the specimen to the optical axis is performed with the controls in the box labeled "Detector". The selected image gas can be admitted or removed and the temperature of the specimen may be changed by the remote user.

atom probe field ion microscope or a three-dimensional atom probe so that they can be monitored or controlled over the Internet. Experiments have been performed that have demonstrated the feasibility of remotely monitoring and controlling a conventional atom probe field ion microscope and a three-dimensional atom probe. However, a significant increase in the effective data transfer rate over the Internet is required to make remote control entirely practical.

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