



Novel Evaporation Control Concepts

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CAMECA Motivation for Improvements in Instrument Control

- 1. Specimen survivability (analysis yield) for complex specimens is less than 100%
- 2. Advanced users often implement manual instrument control methods to improve yield
 - Manually adjustment of data rate during difficult-to-analyze regions
 - Adjustment of laser pulse energy and/or base temperature to reduce evaporation field
 - Modify voltage and/or rate in anticipation of rapid local changes in specimen evaporation based on experience (e.g. previous failures)
- Current instrument control schemes do not necessarily utilize the full range of information available to optimize yield
- 4. Might a modified instrument control scheme lead to a better atom probe? Can we optimize tradeoffs between yield and data quality during the length of an analysis?

Guiding principle for maximizing specimen survivability:

 Reducing the evaporation field for a given specimen increases chance for analysis success



E. A. Marquis et al., Journal of Microscopy 241(3) (2011) 225





Outline

- Current instrument control scheme
 - Voltage control to achieve desired data rate
 - Primary feedback metric is mean detected events per pulse collected across entire detector area
- Examples and ideas for discussion
 - Improvements to voltage control algorithm
 - Constant ion flux
 - Constant charge-state-ratio
 - User-defined control

Discussion



APT Instrument Control Voltage variation to maintain target data rate

Basic Algorithm

- Voltage is changed to keep detection rate (DR) near chosen target (proportional control)
- Adjustable parameters dictate frequency and size of adjustments based on difference between target and actual DR

Motivation

- Ease of implementation
- Provides relatively constant evaporation field for homogeneous materials
 - Imaged tip area generally increases with voltage

Criticisms

- Many materials being analyzed are not homogeneous – field is not constant
- Specimen survival rates (yield) need to improve





Simple Algorithm Modification

Are there any identifiable signals to indicate imminent specimen fracture? Can fracture be avoided?

Yield enhancement possibility

- Short timeframe surges in data rate may indicate increased potential for specimen fracture
- Immediately reduce voltage/rate when these occur to increase survivability

Software implementation

- Analyze response in order to detect rates that greatly exceed the target rate over a small number of pulses
- Immediately tell the hardware to temporarily stop pulsing and lower the voltage when some threshold rate is exceeded

Faster hardware implementation

- Data rates set at 1 event per 100 pulses should rarely experience 9/10 pulses with an event
- Immediately lower the voltage when this occurs





General Background Consider a typical homogeneous specimen

General relationships (Ion Flux)

- Specimens blunt (apex radius (R) increases) during analysis due to non-zero shank angle – imaged tip area increases
- Constant evaporation rate (ER) requires a constant evaporation field (F) → DR and ER are not the same if the radius is changing
- Voltage (V) increases during analysis to offset increasing R
- ER (ions/nm²/s) generally decreases during analysis

Laser pulsing (Temperature and Charge State Ratio (CSR))

- Field is Temperature dependent (F decreases as T increases)
- Heated volumes increase as a specimen blunts leading to lower apex temperatures
- Elemental charge-state-ratio (CSR) for evaporated ions is an indication of F (the higher the proportion of large charge states, the higher the evaporation field)
- Higher CSRs generally indicate increasing evaporation fields for constant laser pulse energy analysis
- CSRs generally indicate tip cooling during analysis





Constant Ion Flux

For a specimen with isotropic field evaporation properties, the following occurs:

- Specimen blunts during the course of the run
- Voltage increases to maintain detection rate
- Imaged area (projection of specimen surface onto detector) increases
- Evaporation rate (ions emitted *per unit area*) decreases

This causes

- Evaporation field decreases over time (reconstruction accuracy degrades)
- Background signal increases over time (decreasing data quality)



Ion Sequence Number

Implementation of constant ion flux correction

- Estimate the surface area change as a function of voltage
- Increase the data rate set point as voltage increases to maintain constant ion flux

Discussion

- Evaporation field increases (relative to standard algorithm) and may promote fracture
- Estimates of surface area become complicated for specimens containing multiple phases



Constant Charge State Ratio

- CSR varies due to both tip shape evolution and apex temperature
- Change laser pulse energy to compensate for changing heated volume (constant evaporation field for homogeneous specimens)

Considerations

- Must have the particular CSR available <u>throughout</u> the analyzed volume
- V and DR are variables that are always well defined
- Flux and CSR depend on estimated evaporation field and atom type respectively – they are not always well defined
- Any algorithms must be prepared to handle ill-defined inputs





Scripted Control

Multiple Control Algorithms

- For an arbitrary specimen/application the user may need to define conditions for changing control algorithms and/or algorithm parameters
- Example: Low field material (A) on high field material (B)
 - Difficulty analyzing through the interface
 - A potential solution is lowering DR while near the interface
- Implementation
 - Track composition
 - Use algorithm 1 with parameters 1a while concentration of A >= 90%
 - At phase transition, when A<90% use algorithm 2 with parameters 2a
 - When B>95% wait 1 million ions and then use algorithm 3 with parameters 3a
- Possible algorithms
 - Voltage control
 - Constant flux Local DRs
 - Constant CSR



