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ATOM PROBE FIELD ION MICROSCOPY OF HIGH RESISTIVITY MATERIALS

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Over the last 30 years, the atom probe has proved to be a powerful tool for studying nanometer-sized compositional fluctuations in a wide range of metallic alloys but has had only limited applications to semiconductors and ceramics.¹ One of the primary reasons for this difference is the higher resistivity of semiconducting and ceramic specimens. Because of this high resistivity, the high voltage field evaporation pulse is attenuated before it reaches the apex of the specimen thereby making the pulse ineffective for field evaporation. Experiments have demonstrated that both variants of the voltage-pulsed atom probe (i.e., those instruments in which the field evaporation pulse is applied directly to the specimen and those in which the negative pulse is applied to a counter electrode in front of the specimen) are equally affected. In this overview, the limits of applicability of the voltage-pulsed atom probe to high resistivity materials are examined.

In this study, a wide range of materials have been examined to determine whether field ion microscopy and voltage-pulsed field evaporation can be achieved and the results are summarized in Table 1. Field ion microscopy including dc field evaporation was possible for all materials except bulk ceramic insulators and glasses. Field ion microscopy requires some conductivity both to achieve a high electric field at the apex of the specimen, and also to support the field ion current. In contrast, voltage-pulsed field evaporation requires transmission of the pulse to the apex of the specimen. All metallic alloys including high resistance alloys and metallic glasses were successfully field evaporated with a voltage pulse. Specimens that were produced from bulk material of several conducting ceramics including MoSi_2 , TiB , and TiC were also successfully field evaporated with a voltage pulse, Fig. 1. In addition, many different types of carbide, nitride, oxide, phosphide and boride precipitates have been characterized in a wide variety of alloys. In the case of semiconductors, high purity silicon ($5 \times 10^3 \Omega\text{cm}$) and SiC were found to be difficult to field evaporate whereas voltage-pulsed field evaporation has been achieved in a lower-resistivity doped silicon ($8 \Omega\text{cm}$).

These results indicate that the borderline of resistivity for voltage-pulsed field evaporation lies in the region of the technologically important semiconducting materials. In general, doped semiconductors with resistivities of 10^{-3} - $10 \Omega\text{cm}$ may be pulse evaporated whereas high-purity semiconductors with resistivities above $\sim 10^3 \Omega\text{cm}$ cannot be pulse evaporated under standard conditions. However, there is no precise "threshold-resistivity" that separates these two regimes. Pulse attenuation does not depend solely on resistivity, but also on other factors such as the geometry of the needle-shaped specimen, in particular the length and taper angle. Long and slender specimens should be avoided. Coating specimens with a conductive layer may partially alleviate this problem. For example, voltage-pulsed evaporation could not be achieved from a Hi-Nicalon (type S) silicon carbide specimen of relatively low resistivity ($10^{-1} \Omega\text{cm}$), fabricated from $10 \mu\text{m}$ diameter fiber or from a $3\text{-}\mu\text{m}$ -thick metallic glass ribbon.

It should also be noted that when typical pulse lengths (10-20 ns) are used with high resistivity materials, there may be an uncertainty in the amplitude of the pulse reaching the specimen apex. This uncertainty may result in preferential evaporation or retention of one or more elements which, in turn, may lead to misleading composition determinations in marginal materials. Voltage-pulsed field evaporation has been reported for high-purity silicon specimens with resistivities as high as $9 \times 10^3 \Omega\text{cm}$, by the application of longer (80 ns) evaporation pulses, and preparing the specimens with a large taper angle.² However, under conditions where the pulse is sufficiently long to allow a significant fraction of the pulse amplitude to reach the specimen apex, a degradation in the mass resolution may be expected, due to an increased uncertainty in the time-of-flight measurements. As a rough guideline, it appears that special care should be taken to ensure reliable data when analyzing materials with resistivities above $\sim 10^{-2} \Omega\text{cm}$ in a voltage-pulsed atom probe.³

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1. M. K. Miller et al., *Atom Probe Field Ion Microscopy*, Oxford, Oxford University Press (1996).
2. A. J. Melmed, et al., *Appl. Phys. Lett.* 39 (1981) 416.
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Table 1. The suitability for field ion microscopy and voltage-pulsed field evaporation of several different classes of materials.

Material	Resistivity Ωcm	Field ion microscopy	Voltage-pulsed field evaporation
Ceramic and glass insulators	$10^{10}\text{--}10^{20}$	no	no
Aluminum oxide/sapphire	10^{15}	no	no
Semiconductors	$10^{-3}\text{--}10^6$	yes	yes/no
Silicon carbide	$10^2\text{--}10^6$	yes	no
High purity silicon	$10^3\text{--}10^5$	yes	no
Doped silicon	$10^{-3}\text{--}10$	yes	yes
Ceramic high Tc superconductors	$10^{-4}\text{--}10^{-3}$	yes	yes
YBCO, BSCCO	$(2.5\text{--}3.0) \times 10^{-4}$	yes	yes
Conducting ceramics	$10^{-6}\text{--}10^{-4}$	yes	yes
Molybdenum disilicide	2×10^{-5}	yes	yes
Titanium diboride	$(0.9\text{--}2.5) \times 10^{-6}$	yes	yes
Tantalum monocarbide	8×10^{-6}	yes	yes
Metals and metallic alloys	$10^{-6}\text{--}10^{-3}$	yes	yes
Metallic glasses	$(0.5\text{--}3) \times 10^{-4}$	yes	yes
Nickel-chromium alloys	$(1\text{--}1.5) \times 10^{-4}$	yes	yes
High alloy steels	$(5\text{--}10) \times 10^{-5}$	yes	yes
Low alloy steels	$(2\text{--}4) \times 10^{-5}$	yes	yes
Carbon steels	$(1\text{--}2) \times 10^{-5}$	yes	yes
Aluminum alloys	$(3\text{--}6) \times 10^{-6}$	yes	yes
Copper	1.7×10^{-6}	yes	yes

FIG 1. Field ion micrographs of MoSi_2 , TiB_2 and Si obtained with the use of neon as the imaging gas.

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