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LASER IONIZATION OF SPUTTERED ATOMS: TRACE ANALYSIS OF SAMPLES WITH ATOMIC DIMENSIONS

(Technical Report)

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Laser ionization of sputtered atoms: Trace analysis of samples with atomic dimensions (Technical Report)

Many technologically important problems involve trace analysis of samples containing only a few atoms. These problems are particularly difficult since they present the simultaneous requirements of efficient sample utilization (high useful yield) and good discrimination (trace impurities). For many analytical techniques these requirements are mutually exclusive. Recent experimental work using laser ionization of sputtered secondary neutral atoms has demonstrated that this technique can achieve part per trillion detection efficiencies with useful yields in excess of 1%. This paper will review and assess this new technique.

I. INTRODUCTION

The need to analyze trace constituents of samples containing only a few atoms is at once an important and a difficult problem. The importance arises from increasingly stringent technological problems exemplified by, but not limited to, the semiconductor industry. The difficulty arises from the need to combine excellent discrimination, in order to identify the trace impurity of interest, with efficient sample utilization. An additional complication often present is the need to limit the analysis information depth to single, surface layers. With surface sensitivity and careful stepwise removal of atomic layers, a depth profile of the trace constituents concentration may be measured.[1]

Consider the analytical problems facing the semiconductor industry over the next decade. Ultra Large Scale Integrated Circuits (ULSI) currently have device features as small as 0.5 µm. As device sizes shrink, both laterally and in depth, entire functional units may be contained in volumes as small as 1 µm x 1 µm x 20 nm containing as few as 2 billion (2 x 10^9) atoms. On such devices as few as 1 impurity atom may be detrimental to device performance.

Small sample analysis capability may also be important in the medical community. Often sample size is limited by the difficulty and damage associated with sample acquisition. Even where large samples may be drawn, benefits such as ease of collection and convenience can be found for efficient sample utilization. In a neutral synapse, for instance, there exists only 50 Na atoms. Thus, single-cell analysis requires many of the same atomic scale sample problems detailed for the case of semiconductor materials.

Historically, Secondary Ion Mass Spectrometry (SIMS) has been the technique of choice for trace analysis of atomic scale samples - particularly for problems associated with surface analysis. SIMS can provide > 1 part per billion (ppb = 10^{-9}) analysis sensitivity for favorable impurity/matrix combinations.[2-9] For trace analysis of atomic scale samples, however, SIMS analysis remains difficult. The useful yield (defined as atoms detected/ atoms removed) of the SIMS technique is often low[2,10,11] because the secondary ion fraction of the sputtering process is small. Generally, the largest fraction of sputtered species (except, for example, alkali metals sputtered from a high work function matrix) are neutral[12,13] ground state[14] atoms. Even for samples with strong ionic bonding half of the sputtered particles are neutral and atomic[15,16]. An additional complication for SIMS analysis is the variability of the secondary ion yields. The secondary ion fraction is strongly dependent upon the element of interest,[2,12-14] the matrix in which that element is embedded[2,18-20] and the chemical state of the matrix surface.[21-24] Moreover, the secondary ions tend to have broad energy distributions and wide angular distributions,[19] leading to low transmission factors for most mass spectrometers.[25]

Many variations of Secondary Neutral Mass Spectrometry (SNMS) are currently being employed[10,11,26] in order to address this problem. Here, the secondary *neutral* atoms are detected usually following post-ionization. Many postionization techniques are being evaluated for SNMS. These include electron beam,[16,27,28] hot electron gas,[29,30] thermal,[31,32] and glow discharge mass spectrometric[33,34] post-ionization. For analysis of atomic scale samples, it is clear that laser post-ionization represents the best combination of efficient sample utilization and discrimination.

The many different methods of laser postionization SNMS[35-61] show impressive detection sensitivity and accompanied many times by high useful yields. In this report the various implementations of laser ionization SNMS will be reviewed and critically analyzed with a specific emphasis on small scale trace analysis.

II. EXPERIMENTAL STUDIES

In this section a brief discussion of experimental sequence for laser postionization SNMS will be presented. While admittedly generalized to encompass all forms of this technique, it is hoped that what follows will give the reader a general impression of laser postionization methodology. Detailed discussions can be found in Ref. 25. Because analysis of atomic dimension samples critically depends upon useful yield, a extended discussion of the various parameters of the useful yield will follow. Finally, an examination of a wide variety of different measurements will be reviewed.

II.1. Experimental design

In principle, a laser ionization SNMS apparatus is extremely simple. The apparatus itself must be ultra-high vacuum since data acquisition times can last up to an hour for the most sensitive analysis. Since these measurements require less than a monolayer equivalent of primary ion dose, sample contamination control is a necessity.

The experimental sequence is initiated when a pulse of primary ions, typically ≅ 5 keV Ar^+, is produced and directed at the target of interest. Depending on the precise measurement being made the primary ion pulse may last from 0.3 ns to 6.0 µs. Shorter pulses are typically used when useful yield is of prime importance.[55,57] Longer primary ion pulses are used for measurements which emphasize detection sensitivity.[40,43] The second step in the experimental sequence occurs following a delay of 100 ns to 1 µs. This delay allows the secondary neutral atoms to fill the ionization region in front of the target. It also gives ample time for rejection of secondary ions and backscattered primary ions which would otherwise represent the bulk of experimental noise in a measurement.[54] Figure 1 depicts the target and a laser postionization region.

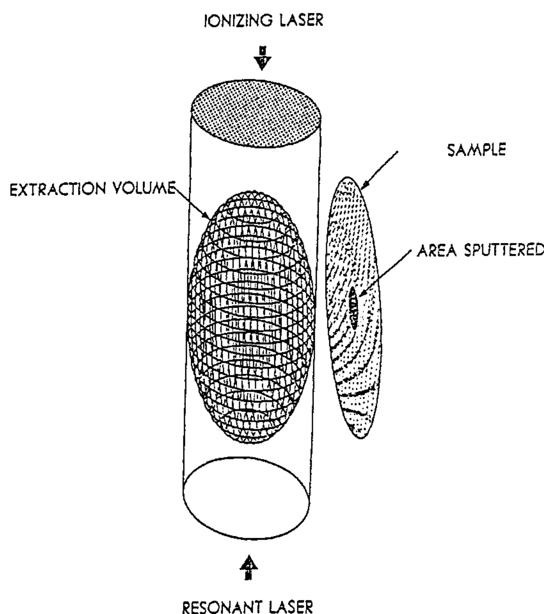

Fig. 1

A three-dimensional view of the active laser ionization volume. Secondary neutrals are ionized along the entire volume illuminated by the laser; however, ion optics considerations limit the extractable ion region to a volume similar to the one shown.

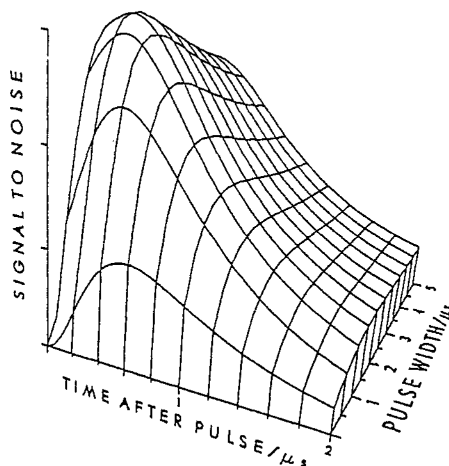

Fig. 2

A three-dimensional graph demonstrating the signal to-noise ratio dependence of a laser ionization SNMS instrument as a function of primary ion pulse width and time delay after the primary ion pulse ends and before the laser fires.

Following ionization by a laser flash, the secondary photoions are extracted and mass analyzed by one of a wide variety of mass spectrometers. Among the mass spectrometers which have been successfully used are quadrupole,[45] magnetic sector,[36,58] and time-of-flight (TOF) systems. Among the various implementations of TOF systems are straight tube-pulsed drawout,[46,62] reflectron[40-49,60,61] and Energy and Angle Refocusing TOF (EAR-TOF).[37-39,50-57]

The design of the laser ionization SNMS is crucial to its operational capabilities. In particular it is useful to consider the interaction between machine design and useful yield.

II.2. Useful yield

The useful yield of all laserbased SNMS systems depends on three parameters. The first parameter is the fraction of sputtered particles which are present in the extractable laser volume during the laser flash. The second parameter is the fraction of those particles in the extractable volume which the laser actually converts to photoions. The final parameter is the transmission of the mass spectrometer.[50] Let us examine each of these parameters in turn.

II.2.1. *Volume density*

Consider the laser ionization volume shown in Fig. 1. The fraction of sputtered particles which enter the laser volume depends on the number density velocity (or equivalently energy) distribution of the sputtered particles of interest and on machine parameters. Theoretical maximums for this tend to be about 10% of the total sputtered flux may be in this volume.[53,54,59]

The interplay of machine design parameters can be seen in Fig. 2. Here, we have assumed that the particles of interest sputter as predicted by linear collision cascade theory.[63-65] A second crucial assumption is that the primary sources of noise are related to the number of primary ions striking the target. Figure 2 shows the signal to noise as a function of primary ion pulse width and of the time delay between the end of the ion pulse and the laser flash. It is instructive to examine the limits of this figure. For arbitrarily short laser fire time delays, no sputtered particles have reached the laser volume. Consequently, no measurement is possible. Similarly, very long primary ion pulse widths do not add to the signal since the sputtered particle density reaches a steady state. However, since by assumption the noise continues to increase, the signal-to-noise drops to zero for long ion pulses.

In summary, a large fraction (10%) of all sputtered atoms are available for photoionization. There is a weak mass dependence to the optimal laser firing delay time. Finally, a complete three-dimensional analysis is necessary to calculate optimum parameters.

II.2.2. *Fraction ionized*

The fraction of sputtered particles photoionized depends on electronic excitation and the method of laser ionization. Resonance ionization requires tunable laser sources and utilizes large resonant cross sections to achieve unit ionization efficiency even for very large laser volumes.[64] The impurity element of interest must be sputtered as an atom in a single electronic energy levels for this method to be effective, however. While this is most often the case, anomalous situations do exist and must be addressed.[62] This situation is similar to matrix effects in SIMS. Conducting samples seem to show less than 10% variation in this regard.[55]

Nonresonant multiphoton ionization is a low cross-section process. However, intense fixed frequency laser sources can, for small laser volumes, reach saturation.[41,42] In contrast to resonance ionization, ground and excited state atoms and molecules ionize reducing matrix effects somewhat. Useful yields are low for this method because the small laser volumes employed limit the volume density reached.[58,60]

While this review has thus far centered upon useful yield as a prime concern, it is important at this point to examine noise generation. In combination, useful yield and noise equivalent signal determine the minimum detectable concentration level. In a well-designed system secondary ion and backscattered primary ion noise sources may be eliminated.[37-41] The primary noise source remaining is the photoionization of isobaric molecules and atoms. This problem is exacerbated by the relatively low mass resolution of most laser postionization instruments.

In the case of nonresonant ionization, this represents a difficult problem which can be ameliorated somewhat by the use of alternative fixed frequency colors.[56] For resonant ionization, the use of additional resonances has been shown to be effective in suppressing nonresonant ionization of isobaric interferences without decreasing the laser ionization efficiency of the impurity of interest.[56,57,64,65] Table 1 demonstrates this increase in detection sensitivity for the use of additional resonances. Explicitly for ^{56}Fe/Si, two results from two different ionization schemes are shown. Figure 3 details a three-color resonant ionization scheme for the Fe atom.

Detection of ^{56}Fe impurity in a Si matrix is an important (Fe is a deep level impurity) and difficult problem. The difficulty arises from the isobaric overlap of ^{56}Fe, the main Fe isotope, with Si_2 the most prevalent molecule sputtered from Si. While the best available SIMS detection limits are in the 100 ppb range,[56] laser photoionization with one resonant photon achieved a detection sensitivity of 2 ppb.[35] This detection

Table 1. Various laser ionization SNMS instruments are compared as to their minimum detection level and useful yield. ppt = 10^{-12} (part per trillion); ppb = 10^{-9} (part per billion); ppm = 10^{-6} (part per million)

Impurity/ Matrix	Detection Limit	Useful Yield	Reference
In/Si	9 ppt	0.0056	40,43
^{54}Fe/Si	34 ppt	0.051	56
In/Si	2 ppb	0.008	36
Os/Ni	4 ppb	0.01	57
^{56}Fe/Si	80 ppt/2 ppb	0.051/0.055	56/35
Si/GaAs	20 ppb	0.0001	58
B/Si	100 ppb	-	60
Ti/Al-Ti-Nb	30 ppm**	-	47
*		0.10	52,54
*		0.001	59

*Theoretical estimates
**Limited by C4 isobaric interference

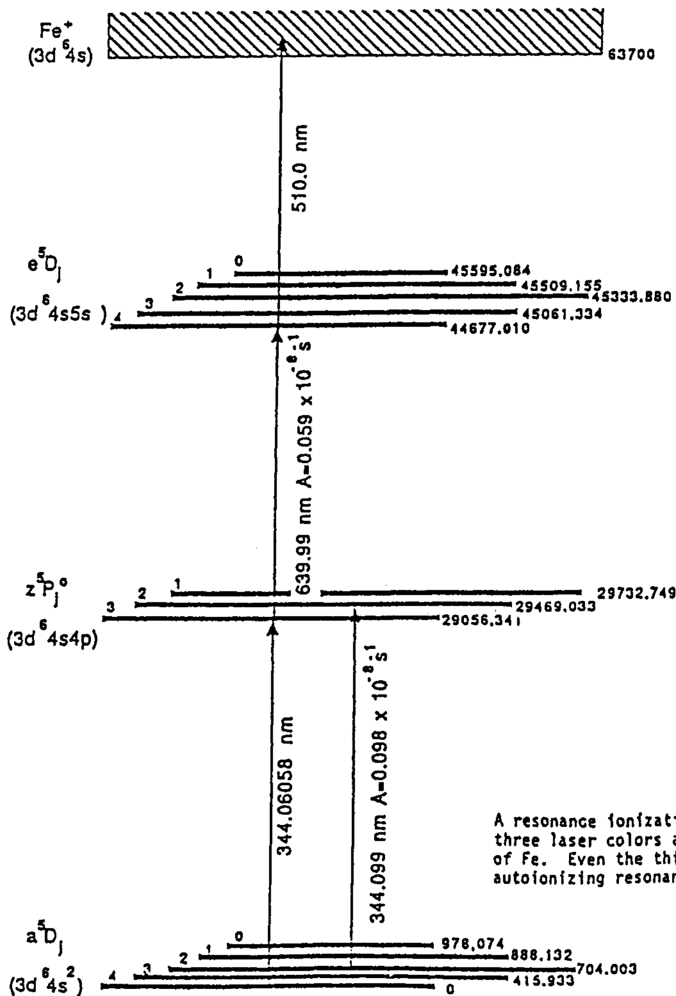

Fig. 3
A resonance ionization scheme for Fe. Note that all three laser colors are resonant with an energy level of Fe. Even the third photon at 510.0 nm finds an autoionizing resonance efficiency.

sensitivity level was limited by residual nonresonant ionization of the Si_2 sputtered flux. By using light from three separate tunable laser sources, a *three-resonant photon ionization scheme pushed this detection* sensitivity to 80 ppt.[56] The power of this technique can be seen from a measurements on ^{54}Fe which has no isobaric overlap with Si molecules. The detection level for this atomic species was 34 ppt only moderately lower than the ^{56}Fe result.[56]

II.2.3. *Mass spectrometer transmission*

The transmission of mass spectrometric systems depends on many parameters. Of particular note here is the difficulty associated with acceptance of a large phase space of photoions. Laser ionization is a volume ionization method. This implies that extracted photoions will have broad energy and angular distributions. Because of the complexity of determining mass spectrometric transmissions, a detailed discussion is not possible, but detailed discussions can be found elsewhere.[25, 52]

Table 1 details various laser photoionization mass spectrometric methods and their associated useful yields. Reference 36 details a magnetic sector mass spectrometer. While the useful yield is lower than can be found for TOF spectrometers, this system is particular useful for isotopic measurements,[58] due to a superior abundance sensitivity. No quadrapolar systems are shown in Table 1. In general, these have inferior properties. All of the results in Table I, except for the In/Si result, were acquired using TOF instruments. As can be seen from the table entries, the useful yields are impressive. They also depend in detail upon the particular TOF system used.[35,40,43,47,56,58,60] Even for the same TOF system constant, useful yields may be found only if the laser ionization process is saturated.[35,56,57]

II.3. Experimental results

Table 1 enumerates various experiments performed with laser SNMS. It includes the impurity measured and its matrix. From the reference indicated in Table 1, the detection limit of the measurement and its useful yield have been distilled. The first two table entries demonstrate the phenomenal sensitivity of the resonant ionization TOF methods. The implication of these two results is that impurities at the ~10 ppt level can be measured in samples with as few as 20 impurity atoms present.

The third and sixth entries in Table I represents the lowest detection level yet achieved by a laser ionization/magnetic sector machine. The useful yield and detection sensitivities are satisfactory but not as good as TOF systems. As stated above, however, the isotopic abundance sensitivity is superior for this machine. This is particularly important for problems of radiochemical dating of both terrestrial and extra-terrestrial samples.

The fourth entry in Table I, Os/Ni, uses *three-color photoionization of Os for detection*. The relatively low useful yield in this measurement arose from an inability to saturate the Os transitions. This case is particularly interesting from two points of view. First, this analysis of a sample of geologic interest was accomplished despite heavy isobaric interference from sputtered molecular constituents. Second, Os is representative of a class of high-ionization potential elements that are particularly difficult to analyze using the SIMS technique. This difficulty arises from the anomalously low secondary ion fraction for these elements sputtered from Ni and other matrices.

The fifth entry in Table I has been described in detail above. It is clear that the use of three-color ionization can provide a substantial improvement in immunity from isobaric overlaps. Moreover, this immunity does not come at the expense of useful yield. Rather, the expense is one of experimental complexity.

The seventh and eighth entries in Table I are nonresonant ionization studies. They were chosen to demonstrate the problems and advantages of this method. All elements are detected simultaneously with similar useful yields. This result is particularly impressive in the case of GaAs where both Ga and As are detected in 1 to 1 ratio.[61] SIMS, on the other hand, shows large variation in the relative Ga and As yields in either positive or negative ion spectra.[10] In favorable cases, such as B/Si, detection sensitivities may reach 100 ppb. Finally, molecular isobaric interferences are almost always limiting. Consider Ti/Al-Ti-Nb.[47] Here, C_4 was present in such abundance and with such a favorable photoionization cross section that detection sensitivity was limited to 30 ppm.

The final two entries in Table I are theoretical estimates of the possible useful yields in resonant laser postionization SNMS. While the published values are two orders of magnitude different, detailed examination shows that the major discrepancy lies in an estimate of photoionization efficiency. For unit photoionization, 0.10 useful yields are possible. For difficult to photoionize elements, such as H, the second estimate is more likely.

III. CONCLUSIONS

Laser postionization SNMS now represents a viable method for trace analysis of atomic scale samples. Typically useful yields near 1% are found for most elements. For this useful yield, detection sensitivities at or below the ppb level (1 in 10^9) are found. To put this in perspective, this implies that 1 ppb impurities may be detected in samples containing as few as 10^{11}atoms. It is clear that resonance ionization is a general process encompassing all elements of the periodic table.[64]

Measurement of such samples still represents a cumbersome, expensive task often requiring the combined use of three dye lasers and ultrahigh vacuum technology. This major impediment to wide-scale use of this technique may be alleviated with the development of diode-pumped solid-state lasers and widely tunable solid-state lasers. Until then, use of this technique will be limited to a few important and difficult problems.

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