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USING HEAVY ION BACKSCATTERING SPECTROMETRY (HIBS) TO SOLVE INTEGRATED CIRCUIT MANUFACTURING PROBLEMS

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Abstract

Heavy Ion Backscattering Spectrometry (HIBS) is a new IBA tool for measuring extremely low levels of surface contamination on very pure substrates, such as Si wafers used in the manufacture of integrated circuits. HIBS derives its high sensitivity through the use of moderately low energy (~100 keV) heavy ions (e.g. C^{12}) to boost the RBS cross-section to levels approaching 1000 barns, and by using specially designed time-of-flight detectors which have been optimized to provide a large scattering solid angle with minimal kinematic broadening. A HIBS User Facility has been created which provides U. S. industry, national laboratories, and universities with a place for conducting ultra-trace level surface contamination studies. A review of the HIBS technique is given and examples of using the facility to calibrate Total-Reflection X-ray Fluorescence Spectroscopy (TXRF) instruments and develop wafer cleaning processes are discussed.

DISCLAIMER

Introduction

As the minimum feature size for integrated circuits decreases and the functional density or number of interconnected devices increases, heavy metal contamination is of concern because it can lead to degradation of device properties, such as gate oxide leakage current or breakdown voltage, which are detrimental to device operation and limit wafer yield.[1,2] Despite this concern, there is an increasing use of heavy metals in semiconductor fabrication, such as those used for interconnects, ferroelectrics, and high-k dielectrics.[1-4] As a result, it has become increasingly important to the semiconductor industry to have surface analytical techniques available with the sensitivity needed to detect, identify, and quantify heavy-metal contamination arising from wafer processing. In response to this need Sandia National Laboratories has recently opened a Heavy Ion Backscattering Spectrometry (HIBS) Users Facility, providing U.S. industry, national laboratories, and universities with a place to conduct ultra-trace level surface contamination studies.

User Facility Description

The HIBS User Facility consists of four main elements and has been described in detail elsewhere.[5,6] The facility includes a small accelerator, analysis chamber, time-of-flight (TOF) detector system as shown in Fig. 1, and data acquisition electronics. The HIBS system was designed to maximize sensitivity to heavy metal impurities that might exist on the surface of semiconductor wafers, and a plot comparing HIBS to Total-reflection X-ray Fluorescence (TXRF) is shown in Fig. 2. Maximizing the sensitivity was mainly accomplished by taking advantage of the Z^2/E^2 dependence of the backscattering cross-section and using a low-energy and high-Z analysis beam (typically, 120 keV C^+). A further gain in sensitivity was also realized by maximizing the detector solid angle through use of a design consisting of three TOF detectors positioned around the incoming analysis beam and having a combined solid angle of ~90 msr. The detectors and their associated foils were placed at optimum angles, as shown in Fig. 3, to help reduce loss in mass resolution due to kinematic broadening.

Wafers are inserted for measurement into the analysis chamber via a load lock enclosed in a class 10 mini-environment. After insertion, a wafer is attached to a four-axis *in-situ* goniometer, allowing the wafer to be positioned for measurement at arbitrary x and y coordinates and for channeling of the analysis beam to reduce multiple scattering background from silicon. As shown in Fig. 3, a low energy C^+ beam is focused onto a spot (~3 mm^2) at the wafer's surface, with the large beam cross section selected to reduce sputtering. It has recently been determined, however, that sputtering is not as serious a limitation to the sensitivity of HIBS as first thought.[7] The backscattered C ions are collimated along their path toward the TOF detectors by apertures in two plates (not shown). This collimation is necessary to eliminate background from stray ions scattering off the chamber or its associated parts. The C ions continue their flight and pass through a thin (~25 nm) C foil, which serves two purposes. Since the overwhelming majority of backscattered C ions are from Si, the first purpose of the foil is to stop these low energy ions that could lead to pulse pileup background. The second purpose is to provide a starting time reference. As each C ion exits the foil, electrons are ejected and accelerated toward an electron multichannel plate (MCP), where they produce a start pulse. Each C ion continues its flight until being stopped by an ion MCP, where it produces a stop pulse. The flight time of the C ion identifies the mass of the near-surface impurity from which the ion backscattered and the total counts in a peak quantifies the composition. Present sensitivity ranges from ~6 x 10^9 at/cm^2 for Fe to ~3 x 10^8 at/cm^2 for Au and is mainly limited by hydrogen multiple scattering from the wafer's surface into the detectors, causing an unwanted and unavoidable background.[6] The mass resolution ranges from ~2 amu at Fe to ~20 amu at Pb. Although the mass resolution is limited, the high sensitivity of HIBS and its survey capability make it ideal for many applications.

Examples

Calibrating TXRF Instruments

Total-reflection X-ray Fluorescence (TXRF) has become one of the most widely used surface analytical techniques for detecting trace levels of surface metallic contamination introduced by semiconductor processing procedures and equipment.[8] The accuracy of the measurements made using TXRF is dependent on the standards used for calibrating each instrument. It is common for TXRF instruments to be calibrated independently and as a consequence, instrument to instrument variations in TXRF calibrations have led to a lack of interchangeability of data, even when measuring the same standards among lab sites within the same company, as reported by Motorola and shown in Fig. 4. This is also true among companies participating in round robins worldwide.[9] This led Motorola to conclude that a universal calibration method was necessary to correct the problem. A four part solution was formulated, which was to 1) use HIBS to measure and verify the accuracy of the TXRF contamination standards used, 2) generate a "universal" sensitivity factor curve using the HIBS certified values obtained from these standards, 3) recalibrate all the TXRF instruments involved in the study using the universal sensitivity factor curve and 4) verify, through another round robin, that new measurements made by the recalibrated instruments are within statistical error. HIBS was selected to verify the accuracy of the standards because it is a highly accurate, sensitive, and non-destructive technique, based on RBS principles. Fig. 5 shows that, except for the Cu measurements, HIBS measurements generally verified the standard vendor's values to within ±10%. Because of the discrepancies in the Cu measurements, the Cu data was not used in the study and only the HIBS areal density values from the Cr, Fe, and Ni standards were used to generate a universal sensitivity factor curve. This curve is used to calculate the reported areal densities from the peak intensities collected during TXRF measurements. The sensitivity factor is determined by dividing the TXRF counts per second (cps) by the HIBS derived areal density (in 10^{10} at/cm^2). For a more complete discussion of how the universal sensitivity curve is generated, see reference [8]. Using the curve, the data from the first round robin, as was shown in Fig. 4, was reprocessed for each of the TXRF instruments with the resulting values reassigned to the respective standards measured as the new "certified" value, even though differing from the HIBS' or standard vendor's value. Then, each instrument was recalibrated using only one of the recertified values, which was necessary because TXRF instruments used in the survey were calibrated with just one element to prevent discontinuities in the calibration curve caused by the software provided by the instrument manufacturer. An example of using this procedure is shown in Fig. 6. The measurements taken during this second round robin, using multielemental standard wafers, generally showed < ±10 % error.

Wafer Cleaning Studies

Research and development efforts to fabricate ULSI dynamic random access memories (DRAMs) have led to the use of heavy metals, such as those used to increase the stored charge in a DRAM cell capacitor. To fabricate these large memory chips, the cell size must decrease and this implies that the area of the cell capacitor must be made smaller, with a resultant decrease in capacitance. Since the charge stored in the capacitor is directly proportional to the capacitance and must be sufficient to send a signal to the sensing circuitry, allowing read out of the cell, a way to increase the capacitance must be found. One way is to compensate for the decreased capacitor area by using high-k dielectric materials to fabricate the capacitor. Examples of materials used for high-k dielectrics are tantalum oxide (Ta_2O_5) and barium strontium titanate ($Ba_xSr_{1-x}TiO_3$) or BST. Platinum is an example of a possible electrode material used in the formation of a DRAM storage capacitor (Pt/BST/Pt).

The use of these metals introduces the issue of how they may be incorporated into a standard wafer fab operation without contaminating the process. To address the issue, several questions must be answered, including 1) how detrimental are the new materials if they cause cross contamination of wafers (especially in the case of Pt as it is extremely mobile in silicon), 2) how robust are current cleaning processes for the removal of these metals from wafers, and 3) what process tools need to be isolated and which ones can be safely shared. To begin answering some of the above questions, wafers were intentionally contaminated with Ba or Pt and then cleaned using various typical cleaning solutions. HIBS analysis before and after the clean (Figs. 7a and 7b) shows that 0.49% HF is effective at removing trace levels of Ba contamination. Typical spectra background can be seen in Fig. 7b, and is caused by both multiple scattering of carbon from the Si substrate and recoiled hydrogen found on the wafer's surface. Metallic Pt removal from a silicon wafer was also demonstrated using a slight silicon etch chemistry in a SEZ RST-201 spin etcher. Physical removal of two microns of silicon in the spin etcher was required to remove the Pt contamination to below 5×10^9 at/cm^2. It is interesting to note that in both cases the HF in the chemical solutions resulted in increased Br on the wafers. This can be an interference for lighter elements but not for ones that are as heavy as Ba or Pt.

Conclusions

HIBS has been shown to be a highly sensitive and accurate tool for the measurement of ultra-trace contamination on the surfaces of semiconductor wafers. Motorola has accepted the HIBS technique to verify the areal density values for their commercially obtained contamination standard wafers. Using HIBS certified values from these standards, Motorola created a universal method for calibrating TXRF instruments. This has improved confidence in the interchangeability of data from different sites within Motorola, and lead to improved quality control. The TXRF calibration method used by Motorola has applicability to the calibration of TXRF instruments world wide. Texas Instruments has also successfully used HIBS technology to evaluate handling and processing of new materials such as Ta_2O_5, BST, and Pt which can be used in the manufacture of ULSI DRAMs. It has assisted in the determination of appropriate cleaning chemistries and processes. Future HIBS work with Texas Instruments will focus on the behavior of low levels of these materials under various manufacturing process conditions.

Acknowledgments

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Figure Captions

Figure 1. HIBS User Facility: A Sandia/Vanderbilt designed time-of-flight detector array, built under a cooperative research and development agreement with SEMATECH, is seen to the right of a sample insertion load lock. The load lock is inside a Class 10 mini-environment. Samples ranging from 1 cm^2 up to 200 mm wafers can be inserted for HIBS analysis.

Figure 2. HIBS vs. TXRF Sensitivities: HIBS and TXRF detection limits for impurities on a silicon substrate are compared. TXRF limits are shown using various x-ray tubes and synchrotron radiation [10].

Figure 3. The HIBS system uses three concentric time-of-flight detectors optimized for large solid angle (~90 msr). A ~3 mm^2 beam spot minimizes sputtering.

Figure 4. Initial TXRF Results: The vendor's certified value for each standard contamination wafer, shown normalized to zero, and the average TXRF value measured for the wafer, obtained from 4 to 6 sites, was used to determine the relative error. The contaminate areal density ranged from ~1 to 6 x 10^{12} at/cm^2. The numbers after the element symbols designate a particular laboratory's wafer set, all of which were single element standards from the same vendor, except for the one marked with an asterisk, which was obtained from a second vendor. "Multi" refers to a multielement standard obtained from a third vendor.

Figure 5. HIBS Results: The vendor's certified value for each standard contamination wafer, shown normalized to zero, and the HIBS value measured was used to determine the relative error. The contaminate areal density ranged from ~4 x 10^{12} to ~6 x 10^{13} at/cm^2. HIBS was used to verify the vendor's standard values at two separate sessions. The second session is noted by an asterisk.

Figure 6. TXRF results, from a representative wafer, show the success of recalibrating four instruments using the HIBS universal sensitivity curve. The reported areal density from each lab was an average of measurements taken at 5 positions on each wafer. The relative error

was determined by comparing the average value from the four labs, shown normalized to zero, with each lab's reported value.

Figure 7. HIBS analysis before a) and after the clean b) shows that 0.49% HF is effective at removing trace levels of Ba contamination. Typical spectra background b) is due to both multiple scattering of C from the Si substrate and recoiled H found on the wafer's surface.

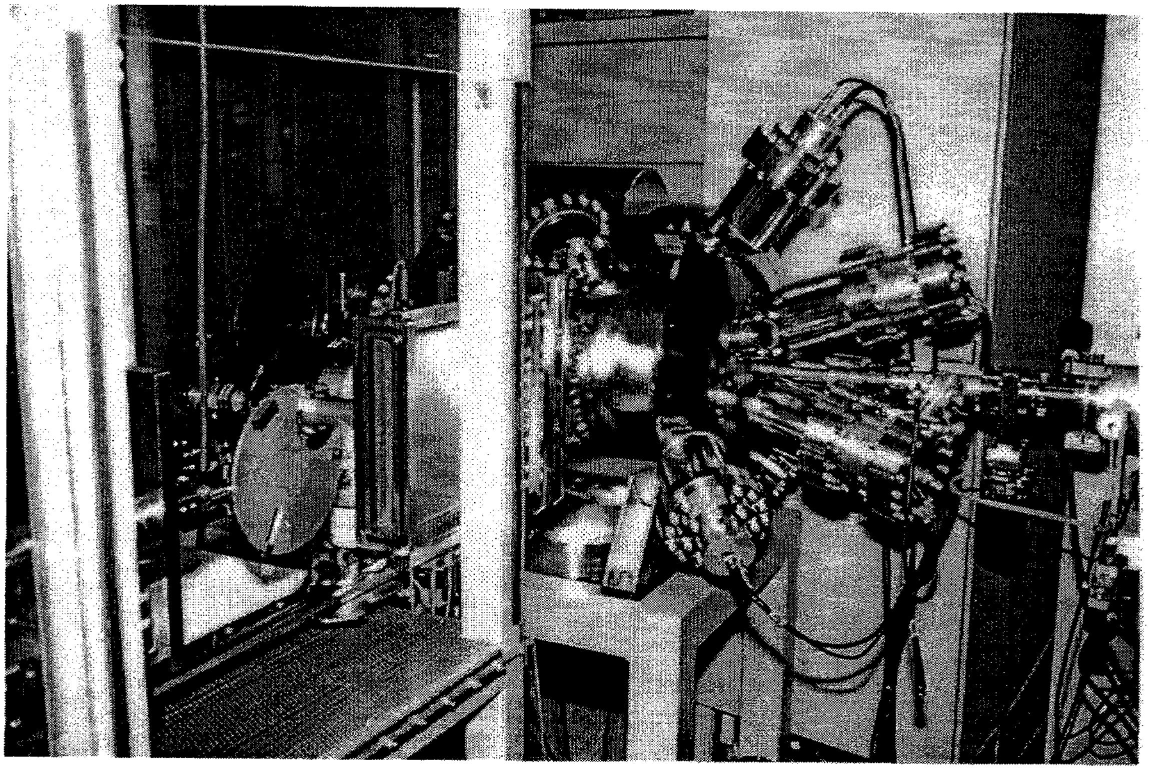


Fig.1

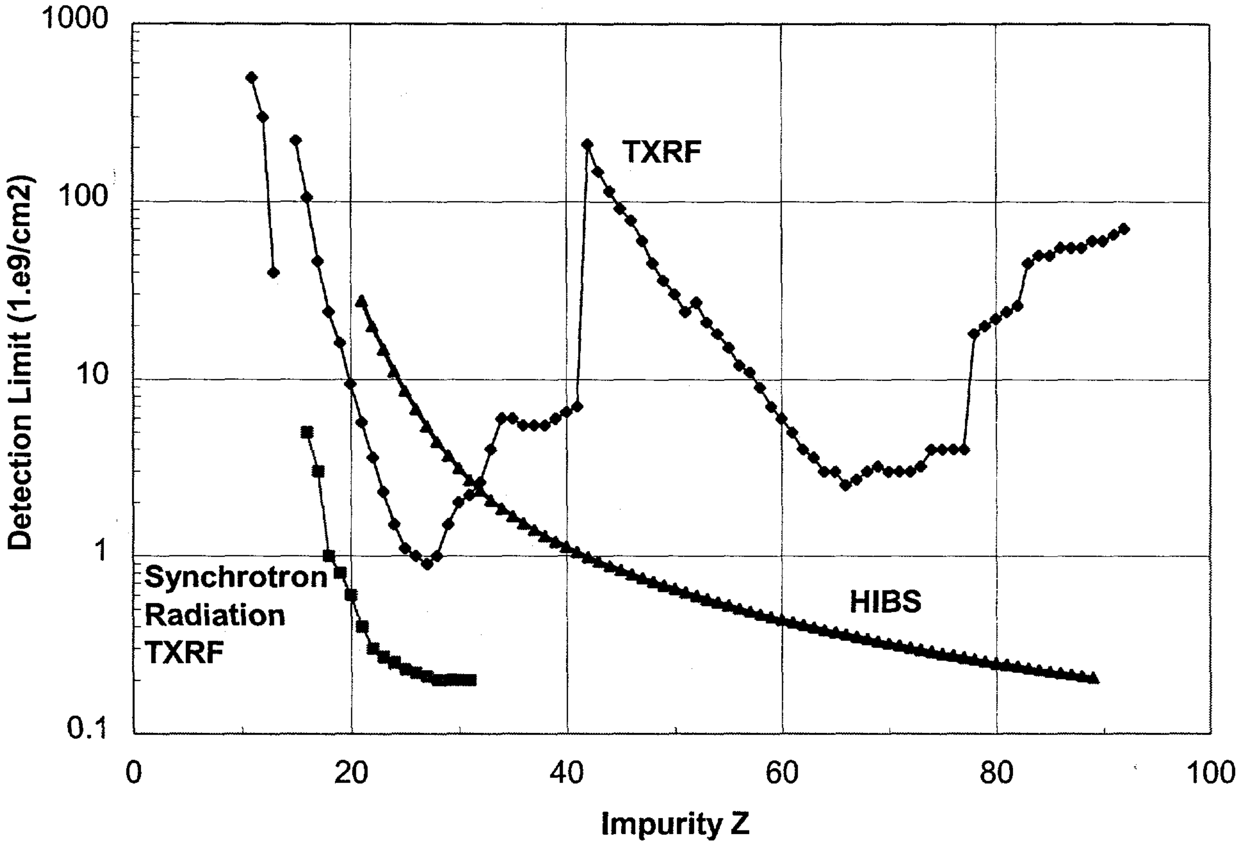

1000
100
10
1
0.1
Detection Limit (1.e9/cm2)
0
20
40
60
80
100
Impurity Z
TXRF
HIBS
Synchrotron
Radiation
TXRF

Fig. 2

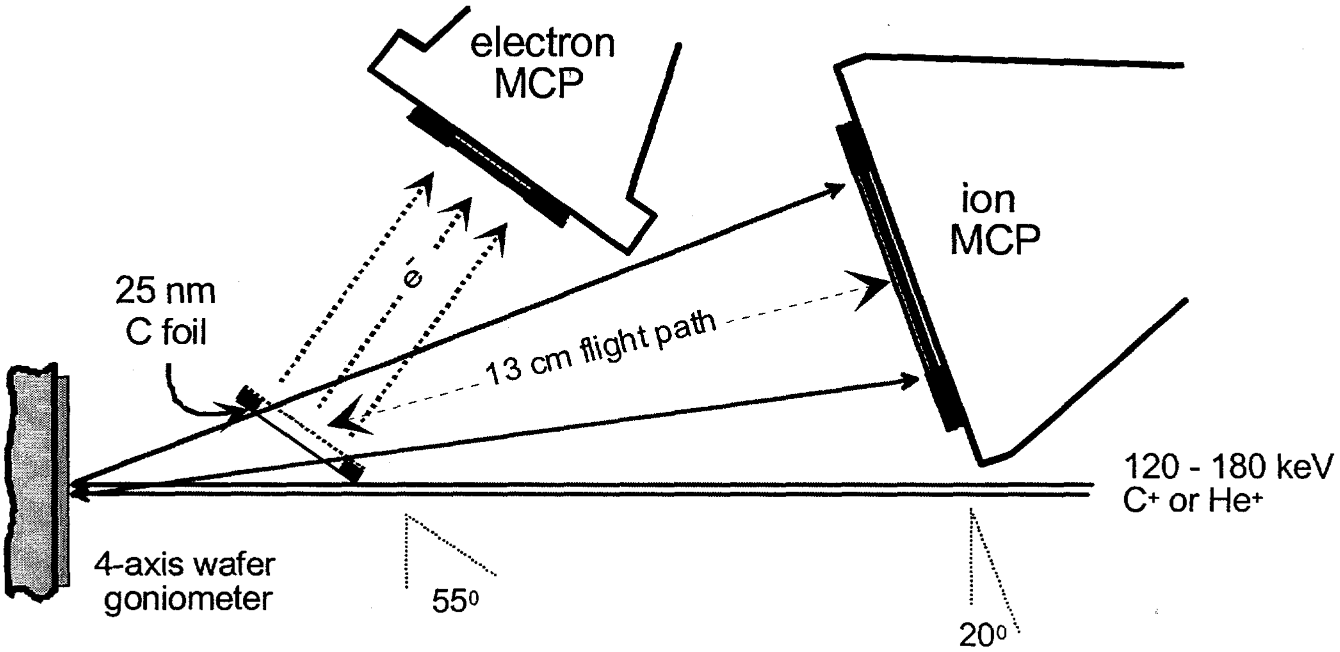
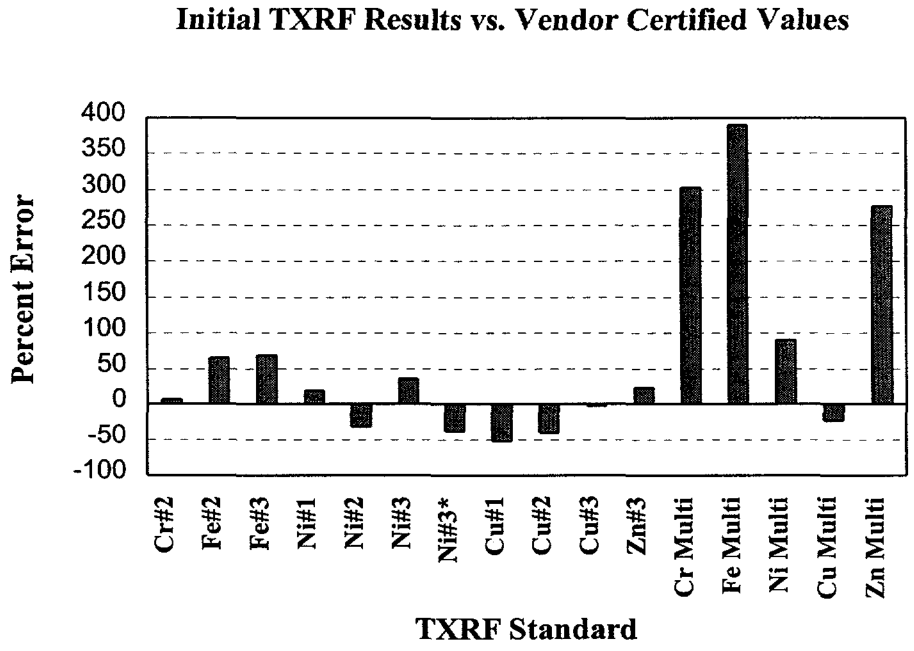
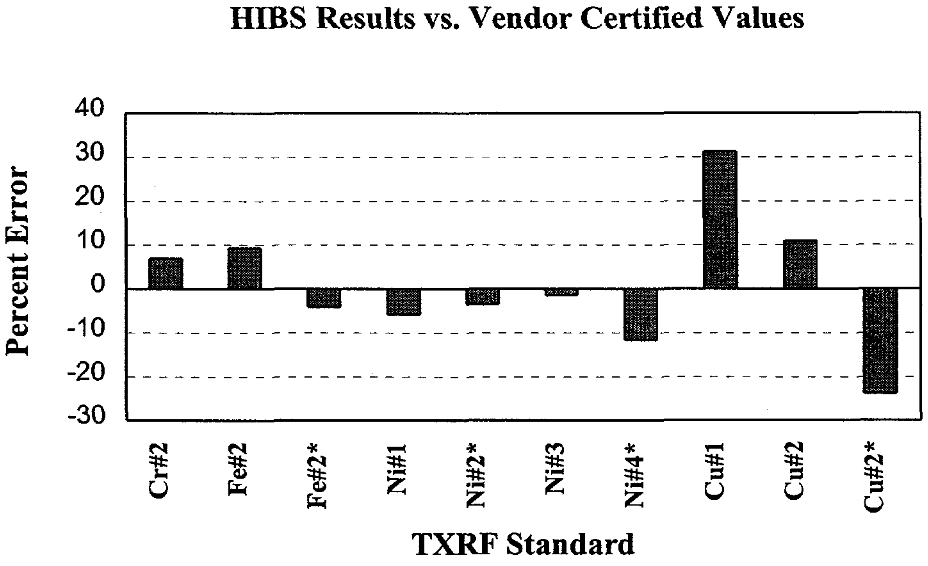

electron
MCP
ion
MCP
25 nm
C foil
e⁻
13 cm flight path
120 - 180 keV
C+ or He+
4-axis wafer
goniometer
55°
20°

Fig. 3



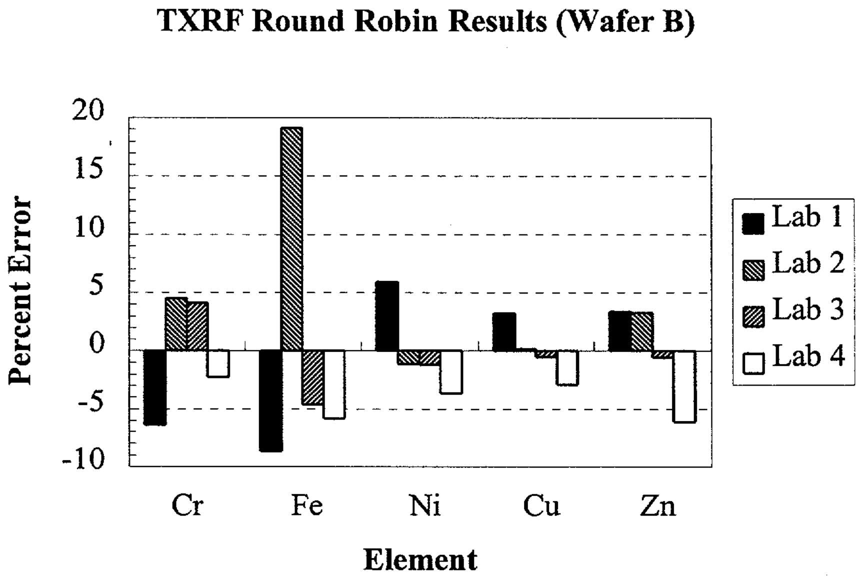
Initial TXRF Results vs. Vendor Certified Values

Fig. 4



HIBS Results vs. Vendor Certified Values
Percent Error
40
30
20
10
0
-10
-20
-30
Cr#2
Fe#2
Fe#2*
Ni#1
Ni#2*
Ni#3
Ni#4*
Cu#1
Cu#2
Cu#2*
TXRF Standard

Fig. 5



TXRF Round Robin Results (Wafer B)

Fig. 6

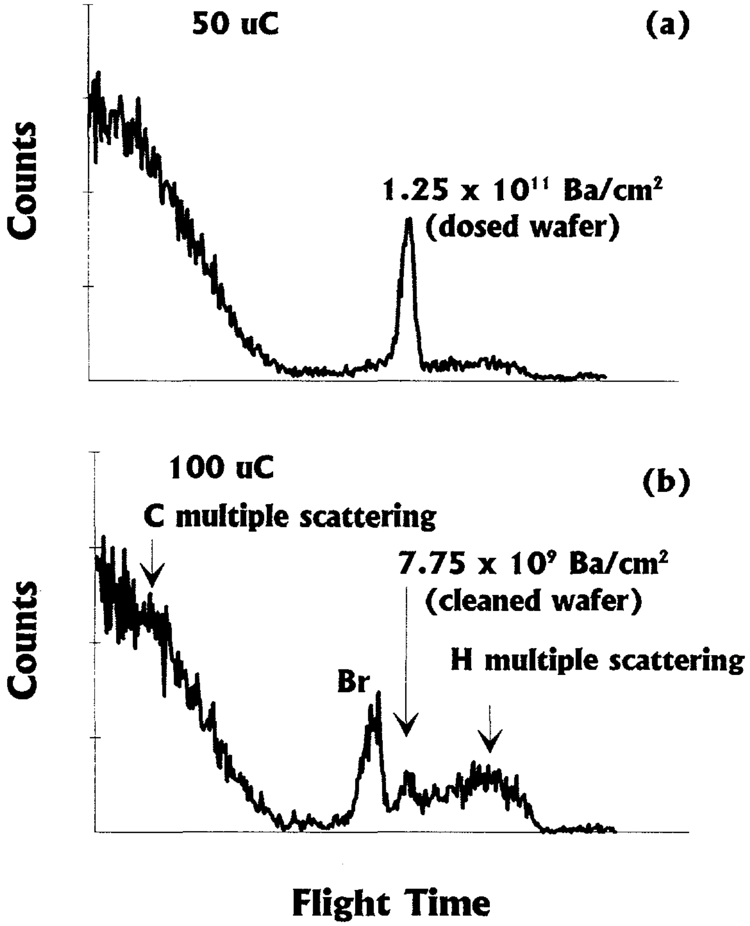

50 uC
(a)
Counts
1.25 x 10¹¹ Ba/cm²
(dosed wafer)
100 uC
(b)
C multiple scattering
7.75 x 10⁹ Ba/cm²
(cleaned wafer)
H multiple scattering
Br
Counts
Flight Time

Fig. 7