

AN UPDATE ON STANDARDS ACTIVITY FOR TXRF AND THE CHALLENGES AHEAD

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ABSTRACT

Standards organizations active in surface analysis using TXRF and VPD/TXRF include: (a) American Society for Testing and Materials (ASTM), (b) Semiconductor Equipment and Materials International (SEMI), (c) Ultra Clean Society of Japan (UCS), and (d) International Standards Organization Technical Committee 201 (ISO TC/201). The standards activities are presently dynamic, and they are on an international scale. This paper provides an update on the status of these activities, and presents the challenges ahead yet to be resolved.

INTRODUCTION

The standardization of TXRF, and VPD/TXRF, for surface analysis of trace metal impurities on silicon wafers is driven not only by their use in qualifying commercial silicon wafers, but also by their use in a broad range of semiconductor applications¹. For example, TXRF is now being used to qualify commercial ion implanters. Equipment companies developing these implanters may have activities in diverse locations in the world: development site, service sites, and, of course, customer sites; so that TXRF measurements may occur on different instruments and different sites on a worldwide scale, and the ability to "get the same answer" becomes commercially critical.

In 1994 the standardization issues were presented in the three main geographic areas which dominate the semiconductor industry: U.S.², UK/Europe³ and Asia Pacific⁴. The standards activities are presently dynamic, and they are on an international scale. This paper summarizes the present status of the worldwide standards activities, and then presents the challenges to be resolved.

PRESENT STATUS OF STANDARDS ACTIVITIES

ASTM

The American Society for Testing and Materials (ASTM) Subcommittee F-1.06 has developed a standard method F 1526⁵ for measuring surface metal contamination on polished silicon wafers using TXRF. In accord with ASTM policy, the approach taken for this standard was to make it broad in the sense of allowing different TXRF instrumentation and measurement configurations. In addition, the method was developed with the intent to update it frequently, as

new information became available. Normally ASTM methods are reviewed every five years for any needed modifications. This test method F 1526 has already been updated twice since 1994. In particular, revisions included a new precision statement based upon recently released round robin data from the Ultra Clean Society of Japan and a new ANNEX discussing the effect of long term repeatability upon estimated detection limits. One consequence of developing the method "early" (ASTM work on the method began in 1990 when this measurement began to impact the semiconductor industry) was that the method for quantification was not resolved. The test method F 1526 avoids this issue and leaves the decision up to the practitioner. One reason for avoiding this issue was the international disagreement on how to perform the quantification.

SEMI

Semiconductor Equipment and Materials International (SEMI) normally does not develop standard test methods. However, in order to meet their timing needs, the SEMI European Equipment Automation/Hardware Subcommittee on Minienvironments introduced a VPD/TXRF test method (SEMI Standards Document E45⁶) as part of their SEMI specification on contamination from minienvironments. This SEMI Subcommittee obtained input from the ASTM Subcommittee F-1.06 during the test method development. However, the SEMI test method does not have a precision statement, and the method is very restrictive in that a practitioner must have a state-of-the-art wafer cleaning bench/process, the minienvironment to be tested, and a TXRF, all located within 10 minutes of each other. This restriction constrains the use of the method to one facility in the world.

In 1995 a SEMI European Silicon Wafer Subcommittee task force began developing a more complete VPD/TXRF test method to support the minienvironment test method.

The SEMI North American Subcommittee on Silicon Wafers has incorporated reference to ASTM F 1526 in SEMI Standards M1⁷ and M18⁸ and recommended, in a non-binding appendix to SEMI Standard M1, maximum levels of metal contamination on polished silicon wafers to be used to manufacture integrated circuits with design rules between 0.8 and 1.2 microns. The ASTM F 1526 is one of the test methods listed to help companies meet the recommendation.

UCS

In 1994 the Ultra Clean Society of Japan (UCS) Working Group on TXRF, an unofficial consortium of silicon wafer suppliers and IC manufacturers, completed two years of experiments designed to determine an acceptable quantification method and to improve the poor round robin results^{9,10} achieved previously in Japan under the auspices of ASTM F-1. The UCS proposed a standard test method based upon their experimental results.¹¹ The UCS also obtained input from ASTM F-1.06 on the standards issues in developing their proposed method. A key difference between the UCS and ASTM approaches to a standard TXRF method is that the UCS proposed a method which is less broad than the ASTM method. For example, the UCS method allows only one type of reference sample.

ISO TC/201

The International Standards Organization Technical Committee 201 Working Group 2 on TXRF (ISO/TC 201/WG2) began work in 1994 on a draft method for TXRF analysis of silicon wafers. They have obtained input from both the ASTM F-1.06 and the UCS. They have plans to complete an international round robin in 1995. At the present there is no consensus standard.

CHALLENGES AHEAD

The challenges ahead are mainly: (1) resolve the issue of reference standards, (2) resolve whether the test method should be restrictive or broad, (3) complete an acceptable international round robin, and (4) reach agreement on relative sensitivity factors (RSFs).

The issues related to reference standards were presented elsewhere.² An alternative approach is being studied by Schwenke and Eichinger¹². The idea is to use the bulk silicon itself as the reference sample, as a solution state fitting with theory as proposed by Schwenke and Knoth.¹³ One advantage of this is that all practitioners in the world could easily obtain equivalent reference samples (polished silicon wafers), and the long term stability of the reference would be excellent (unlike many of the presently proposed reference sample types). The absorption from the detector window could conceivably be handled via the RSFs. One consequence of this approach is that the silicon matrix signal could not be also used to determine the angle calibration or to perform a fine tuning of the angle calibration, practices which some instruments do.

The issue of whether the test method should be restrictive or broad is partly a matter of philosophy, but also a consequence of different laboratories obtaining widely different results when using different instrument configurations. Of course, the different results may have been seriously affected by also using different reference standards, so if the reference standard issue can be satisfactorily resolved, then this issue may go away.

The issue of an acceptable international round robin will follow from resolution of the first two issues. The round robin will most likely be overseen by the ISO TC 201/WG2.

The issue of reaching agreement on RSFs is important, but relatively new. The different manufacturers software use different RSFs even when using the same x-ray source. Furthermore, there are different x-ray sources in use (W, Au, Mo), each with their unique RSFs.

REFERENCES

1. R. S. Hockett, "TXRF Semiconductor Applications," Advances in X-Ray Analysis Vol. 37, edited by J. V. Gilfrich et al, Plenum Press (New York, NY) pp. 565-575 (1994).
2. R. S. Hockett, "TXRF Reference Standards: A Discussion," Contamination Control and Defect Reduction in Semiconductor Manufacturing III, edited by Dennis N. Schmidt, ECS Proceedings Vol. 94-9, The Electrochemical Society, pp. 323-338 (1994).
3. R. S. Hockett, J. M. Metz, and S. Tan, "Quantification Issues for VPD/TXRF," Proceedings of the Second International Symposium on Ultra-clean Processing of Silicon Surfaces, edited by March Heyns, published by Uitgeverij Acco, Leuven, Belgium, pp. 171-176 (1994).
4. R. S. Hockett, "A Review of Standardization Issues for TXRF and VPD/TXRF", Advances in X-Ray Chemical Analysis, Japan, Vol. 26s, pp. 79-84 (1995).
5. ASTM F 1526, "Standard Test Method for Measuring Surface Metal Contamination on Silicon Wafers by Total Reflection X-Ray Fluorescence Spectroscopy," 1995 Annual Book of ASTM Standards, Vol. 10.05, American Society for Testing and Materials, Philadelphia, PA, 1995.

6. SEMI E45, "Specification for the Determination of Inorganic Contamination from Minienvironments," SEMI International Standards 1995, Semiconductor Equipment and Materials International, Mountain View, CA, 1995.
7. SEMI M1, "Specifications for Polished Monocrystalline Silicon Wafers," *ibid.*, 1995.
8. SEMI M18, "Format for Silicon Wafer Specification Form for Order Entry," *ibid.*, 1995.
9. R. S. Hockett, S. Ikeda, and T. Taniguchi, "TXRF Round Robin Results," Cleaning Technology in Semiconductor Device Manufacturing, edited by J. Ruzyllo and R. E. Novak, ECS Proceedings Vol. 92-12, The Electrochemical Society, Pennington, NJ., pp. 324-337 (1992).
10. R. S. Hockett, S. Ikeda, and T. Taniguchi, "Round Robin Results for TXRF," Extended Abstracts Vol. 92-2, Abstract No. 340, p. 498, The Electrochemical Society, Pennington, NJ., p. 498 (1992).
11. K. Kawai, "Standardization of TXRF and Its Measurement Results," Proceedings of the 22nd Symposium on ULSI Ultra Clean Technology, August 29-31, 1994, Tokyo, published by The Ultra Clean Society, p. 406 (1994).
12. Private Communication, Dr. Peter Eichinger, GeMeTec, Munich, Germany, 1995.
13. H. Schwenke and J. Knoth, "Depth Profiling in Surfaces using TXRF," Advances in X-Ray Chemical Analysis, Japan, Vol. 26s, pp. 137-144 (1995).