Y-12

**OAK RIDGE
Y-12
PLANT**

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Y/AMT-454

Final CRADA Report
for
CRADA Number Y-1292-0130

C/Y-12--92-0130

**DEVELOPMENT OF XRMF TECHNIQUES FOR
MEASUREMENT OF MULTI-LAYER FILM THICKNESSES
ON SEMICONDUCTORS FOR VLSI AND ULSI INTEGRATED
CIRCUITS**

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February 3, 1997

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for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

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Abstract

A CRADA with Kevex Instruments was carried out to develop improved XRMF instrumentation for the nondestructive analysis of electronic components during manufacture. Experiments conducted at Y-12 proved the feasibility of a new Kevex x-ray tube design. Tests also show that the current commercial supply of straight glass capillaries is unreliable; however, other vendors of tapered single and multiple glass capillaries were identified. The stability of the Y-12 x-ray microprobe was significantly enhanced as a result of this CRADA.

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Objectives of the CRADA

The goal of this project was to develop photon-induced x-ray microfluorescence (XRMF) techniques for nondestructive, high-spatial resolution measurements of the thicknesses of thin films on semiconductors for the manufacture of VLSI (Very Large Scale Integration) and ULSI (Ultra Large Scale Integration) circuits. The new XRMF techniques would also be applied to the improvement of nondestructive x-ray microanalysis methods for DP materials.

The manufacture of reliable VLSI and ULSI circuits requires extremely uniform film thicknesses. Because VLSI and ULSI circuits are very small, there is a critical need for a thin film measurement capability with spatial resolution less than 10 microns. Photon-induced XRMF is the only method capable of nondestructively measuring the thicknesses of thin films. The Oak Ridge Y-12 Plant has the only non-synchrotron based x-ray source capable of providing spatial resolutions less than 10 microns. Kevex Instruments (formerly Fisons Instruments) currently markets an XRMF spectrometer with a spatial resolution of 100 microns and is heavily involved in developing thin film measurement and quantitative element analysis methods. These methods are currently being developed for on-line semiconductor processing, where higher intensities and small analysis areas are required in the future. Kevex Instruments also has expertise in the design and manufacture of reliable x-ray tubes. MMES, on the other hand, has an XRMF capability in a laboratory-based x-ray microprobe with a demonstrated spatial resolution of 3 microns. The Y-12 x-ray microprobe (Y12XRM) was awarded an R&D 100 award based upon its unique method of attaining high x-ray brilliance.

The Y12XRM has been used for the analysis of inclusions and tracers in special materials, in corrosion and diffusion studies, in the determination of failure modes, and even in the analysis of fish as part of an environmental investigation. The improvements in the Y12XRM as a result of this project were needed to increase reliability, reduce turnaround time, and provide needed new capabilities. The project will benefit DP programs in assuring nuclear competency. The knowledge gained from the project will assist U.S. industry in regaining a position of leadership in the field of electronics by providing the technology to build on-line nondestructive analytical instrumentation to replace time-consuming, expensive destructive techniques.

Discussion as to Whether the Objectives of the CRADA were Met

The objectives of the CRADA were met through knowledge gained in optimizing the design of XRMF instrumentation. Experiments conducted at the Y-12 Plant as part of this CRADA led to an understanding of the limitations of commercially available glass capillaries to be used for conditioning the x-ray beam for XRMF analysis. It was obvious from the data that more stringent manufacturing methods than those currently in use would be required to produce reliable and reproducible glass capillaries for use in manufactured XRMF instrumentation. Other experiments showed that a proprietary

KeveX x-ray tube design was feasible and could produce higher x-ray output than conventional designs. These experiments allowed KeveX to make production decisions affecting their next generation XRMF instrumentation.

How the Work done under the CRADA Benefited the DOE office that Funded the CRADA

The knowledge gained from the work done in this CRADA led to improvements, both actual and potential, in the XRMF instrumentation at the Y-12 Plant. This instrumentation supports Defense Programs by providing analytical support for current tasks and as a prototype for on-line microanalysis in a downsized factory.

Technical Discussion of Work Accomplished

KeveX performed computer modeling of a new anode head geometry. The critical issues were the power loading at the anode (number of incident electrons at a certain energy per unit area of the spot), electron focusing (spot size), the distribution of generated x-ray photons as a function of energy and take-off angle and the distribution of generated back-scattered electrons as a function of energy and take-off angle. The modeling results showed an improvement in the number of x-ray photons generated in the spot as compared to the conventional x-ray geometry.

A preliminary design concept of the new anode head for an x-ray tube was developed. The new electron and x-ray geometries provided for higher power loading density in the smaller area. The design concept was transferred to Y-12 for an actual anode head design, development and manufacture. The anode head was designed, fabricated and tested at Y-12. The tests showed improved count rates and higher target loading capability, indicating that the concept was feasible. The tests also identified a potential problem area requiring further experimentation to find a solution.

Commercially available glass capillaries, used initially in the Y-12 system to guide x-rays from the focal spot to the specimen, were tested at Y-12 for lot variability. Significant variability was detected in capillary diameter. Other suppliers of capillaries were investigated. Although no other manufacturers of thick walled, straight capillaries were found, manufacturers of tapered single and polycapillary optics for x-ray beam transmission, were found. At least five vendors of tapered single capillaries and two vendors of tapered polycapillaries are currently in business. Some of these capillaries were tested as part of another CRADA.

KeveX applied their expertise in x-ray tube technology in helping Y-12 identify sources of x-ray tube instability in the x-ray microprobe. Based on that input and the input of another non-KeveX group, modifications were made to the Y-12 x-ray microprobe leading to significant stability improvements. In addition, Y-12 made modifications to

the data collection routine which allowed the microprobe to accommodate the remaining instability.

Patents

No patents were applied for as a result of this CRADA.

Assessment of Commercialization Possibilities

Kevex is currently assessing the commercialization possibilities of a new XRFM instrument, based, in part, upon the results of this CRADA.

Plans for Future Collaboration

There are currently no plans for future collaboration.

Conclusions

The objectives of the CRADA were met in that improvements were made to the Y-12 x-ray microprobe and important design and manufacturing decisions were made by Kevex regarding their next generation XRMF instrument. The Y-12 instrument has significantly improved geometry, output, and stability as a result of this CRADA. The next generation Kevex instrument will have improved output as a result of x-ray tube and geometry improvements.

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