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Final Report
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Research at and Operation of the Materials Science Beamline (X-11)
at the National Synchrotron Light Source

Dale E. Sayers
Principal Investigator

North Carolina State University
Raleigh, NC 27695

DOE Patent Clearance Granted
MP Dvorscak
10/29/03
Date
Mark P. Dvorscak
(630) 252-2393
E-mail: mark.dvorscak@ch.doe.gov
Office of Intellectual Property Law
DOE Chicago Operations Office

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General beamline operation and research

This document represents the final report for DOE Contract No. DE-FG05-89ER45384 on "Research at and Operation of the Materials Science Beamline (X-11) at the National Synchrotron Light Source." The X-11 Participating research team (PRT) was founded in about 1978 and was first funded by DOE in 1979 to construct and operate a beamline dedicated to materials science applications of x-ray absorption spectroscopy (XAS). The original team was the prototype of what a PRT was meant to be. It consisted of mixture of university, national laboratory and industrial research groups, each of which contributed to the construction and operation of the beamline. Dale Sayers of North Carolina State University (NCSU) was the PI on the contract and PRT Spokesperson from its inception until the end of this contract but Joe Budnick of the University of Connecticut also played a major leadership role throughout the term of the PRT. Over the years the constitution of the PRT changed over the years. Initially departing

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members were replaced either by new members or by the expansion of time and commitment by existing members. Over about the last five years, new members who could contribute resources were not found. Some of the revenue lost by this was made up for by short-term relationships with several organizations but this was not completely covering the shortfall in operational expenses. At the time that this contract was ended, the PRT was actively pursuing other avenues of support, primarily through a partnership with the NSLS. After the termination decision was made, the PRT was able to partner with the Naval Research Laboratory (NRL), who had been a PRT member, to take over the operation beginning in mid-2001. They have been operating X-11A since then as part of their synchrotron beamline consortium.

Technically, X-11 made many contributions to the field. One of the early goals was to make an operationally robust but user-friendly facility that was optimized for the source. We were the first beamline to really provide such an environment. The operating software was ahead of its time in providing a user-friendly interface with analysis capability available at the beamline. The original code developed at X-11 served as a model for other lines at the NSLS and at other facilities. The PRT also played a lead in developing sagittal focusing and in a number of other technical developments. The operational capability of X-11A was shown from the beginning. It was the first PRT line to operate and had the first publication for data taken on the x-ray ring. It very quickly established a full time users program, first for PRT members and then for general users. We were in the first wave of lines to accept general users on the x-ray ring and have maintained a full general users program from the beginning. Throughout its history X-11 was generally about three times oversubscribed for users because of its reliability and the quality of data it produced. During this operational period we also managed to maintain as much of a development and upgrade effort as time and budget would allow. We essentially were able to keep the capabilities of the line near the state-of-the-art for bending magnet lines on a source of this size. Over the history of nearly 20 years of operation we have a record of more than 95% availability. Productivity has also been extremely high. From the beginning the PRT reported about 40 publications a year including refereed conferences and publications. This has included an active and steady publication record from our general users as well. In the early days of the NSLS x-ray ring operation we were responsible for a significant fraction of the total NSLS publications. Even today, the integrated number of publications from X-11 represents a significant fraction of the total NSLS publications, far out of proportion with most other beamlines. In fact, after having reviewed a number of synchrotron facilities and beamlines, it is clear that we continue to be more productive than most beamlines in the US synchrotron complex.

One of the major reasons that we were able to be so successful was the quality and quantity of our support staff. The total X-11 operational budget was about \$400,000 per year (and essentially flat for its entire existence). About 35% of this amount (\$140,000) was provided from this DOE contract to cover the core university users. The remaining 60% came from the other PRT members. Typically our staffing included a beamline scientist and a technician and a part-

time administrative assistant all located at BNL. At times there were visitors, post docs and students who were able to be in residence at BNL and supported the operation. Although this operational budget is low by the standards of third generation lines, it was high compared to most other PRT's at the NSLS but, we believe, was essential for maintaining our high degree of performance.

One of the additional contributions of X-11 has been to the development of XAS and to the synchrotron radiation community. The XAS community is one of the largest discipline communities in synchrotron radiation. The growth of SR in the US was closely coupled to the development of XAS. The initial results at SSRP in 1974 demonstrated the potential of this "new" field and was one of the major arguments for developing dedicated second generation sources. However, like any new discipline it would take many man-years of development to reach its potential. When the opportunity came for some of the founding X-11 PRT members (Sayers, Stern, Heald, Bunker) to be involved in developing their own dedicated line at the NSLS, they were able to greatly improve on the facilities that were available at that time at SSRL. In addition to some of the technical advances mentioned above, members of X-11 played a number of important roles in developing XAS to the point where it is today. Most significantly, this included the creation of the XAS Standards and Criteria Committee which held its first meeting at the NSLS and later spun off the development of the International XAFS Society. These efforts were the main reasons that the credibility of the field has grown over the past decade. X-11 PRT members have also been active members of the NSLS community including head of the users organization and serving of all of the major NSLS committees. The first XAS workshops were run by X-11 members. We have also served as one of the main training grounds for synchrotron scientists around the world. Many of the scientists, post docs, and students at X-11 now are running their own lines or serving as beamline scientists at synchrotron facilities around the world. A main reason for this is the close coupling and support for university groups with the PRT's. This is likely to be one of the greatest casualties of the change in operational philosophy for PRT's that has been instituted by the DOE in recent years.

The publication record of the PRT was briefly described above. A publication list for X-11 since the time of the last progress report in 1999 is attached to this document as an appendix. It lists nearly 70 publications during this time plus 8 more PhD's awarded on the basis of work done on X-11. As with all previous publication lists it represents quality, quantity and diversity. This latter trait being a particular strength of XAS. A partial list of research areas covered here include semiconductor interfaces, high T_c materials, catalysts, battery and fuel cell electrodes and a number of environmental studies, with publications in the top journals in these areas.

Core Science at NCSU

The scientific effort at NCSU is directed towards the formation of epitaxial films and nanostructures on compound semiconductors, specifically SiC and alloys from the Si-Ge family. Publications resulting from the core research effort are highlighted by an asterisk (*) in the attached appendix. Six papers are included not counting the papers in review or draft described in sections 2 and 3 below. Since the last progress report, progress has been made in three areas of research.

1. TiC nanoisland formation on 6H-SiC(0001)_{Si}

W. Platow, J. Oh, R. Nemanich, D. Sayers, J. Hartman, R. Davis NCSU

Simultaneous formation of titanium carbide nanoislands on silicon carbide substrates has been studied with scanning tunneling microscopy and x-ray absorption near edge spectroscopy. Scratch-free and atomically flat 6H-SiC(0001)_{Si} substrates were prepared by high temperature hydrogen etching. The surfaces were subsequently cleaned by *in situ* ultrahigh vacuum annealing. Titanium carbide nanoislands were formed by titanium deposition and annealing at 950°C. The average width (10-50 nm) and the separation of the Ti islands was controlled by varying the titanium coverage (0.1-0.3 nm) and the annealing time (1-20 min). At the lowest coverage, the islands were uniformly distributed over the surface, while at higher coverage the islands tend to collect at the substrate step edges.

This work was published in J. Applied Physics (91) 6081-6084 (2002).

2. Growth of Thermodynamically Stable Titanium Germanosilicide Thin Films on Strained Si_{1-x}Ge_x

J. Burnette, R. Nemanich, D. Sayers NCSU

The sequential deposition of strained Si_{1-x}Ge_x with concentrations $x = 0.20$ and 0.30 , amorphous silicon, and titanium on Si (100), with anneals at 700° C leads to the formation of a C54 Ti(Si_{1-y}Ge_y)₂/Si_{1-x}Ge_x bi-layer, whose phase formation and interface stability are studied. The use of an amorphous layer of Si is employed to eliminate or decrease the formation of germanium-rich Si_{1-z}Ge_z alloy precipitates found in the solid phase reaction of Ti and Si_{1-x}Ge_x. It has been proposed that the precipitation phenomenon was driven by changes in the enthalpy of formation as a function of concentration in the Ti(Si_{1-y}Ge_y)₂ layer, resulting from the enthalpy difference between TiSi₂ and TiGe₂ compounds, both of which are assumed to be completely miscible with one another. In this study, a Gibbs free energy model parameterized in terms of atomic percentage of Ge was used to determine the concentration of the Ti(Si_{1-y}Ge_y)₂ compound in equilibrium with the Si_{1-x}Ge_x substrate. Layers of amorphous silicon of varying thickness were incorporated between a 300 Å Ti layer and the strained Si_{1-x}Ge_x substrate layer to achieve Ti(Si_{1-y}Ge_y)₂ films that are in equilibrium with the Si_{1-x}Ge_x substrate. The use of amorphous silicon layers of varying thickness indicated that Ti(Si_{1-y}Ge_y)₂/Si_{1-x}Ge_x films could be formed with the absence of germanium rich precipitates at the grain boundaries, depending on the amorphous silicon layer thickness.

This work has been submitted to the Journal of Applied Physics.

3. Titanium Interlayer Mediated Epitaxy of CoSi_2 on $\text{Si}_{1-x}\text{Ge}_x$

J. Burnette, D. Sayers, R. Nemanich NCSU

Titanium Interlayer Mediated Epitaxy (TIME) has been shown to promote the formation of epitaxial CoSi_2 on both Si (100) and Si (001). Because of the emergence of the Si-Ge as an important technological material, a better understanding of the mechanisms of formation of CoSi_2 as a contact material needs to be developed. Structural similarities between Si and $\text{Si}_{1-x}\text{Ge}_x$ alloys have motivated a study of whether TIME could be successful in forming epitaxial CoSi_2 on $\text{Si}_{1-x}\text{Ge}_x$. Titanium layers of varying thickness were deposited as interlayers between Co and c-Si/ Si_8Ge_2 pseudomorphically grown on Si (100) to investigate their role in the formation of epitaxial CoSi_2 on

The effect of Ti interlayer thickness on the selective orientation of CoSi_2 to the $\text{Si}_{1-x}\text{Ge}_x$ substrate, and the circumstances under which a polycrystalline CoSi_2 film has been formed has been studied. We have found that Ti was beneficial in promoting epitaxy, and experimental results indicate that an ideal diffusion barrier thickness can be determined. In addition, it is shown that Ti was active in the formation of its own silicide, in contrast to other reports which suggest that it played a more passive role in the formation of silicides in the Co/Ti bi-layer arrangement.

This work is in draft form and will be submitted for publication shortly.

Final Comments

After more than 20 years of successful operation and scientific productivity, the DOE funding for this operation was terminated so abruptly that it caused considerable problems for the X-11 PRT and the core university supporters that several comments are in order. The termination of this program was done under a new policy to discontinue support of individual PRT's and a transfer of operations to the individual facilities. While the reason for this new policy is understandable in today's funding and research climate, it's implementation in general, and it's impact on X-11 in particular were not done well. When publicly discussing the success of the synchrotron facilities, the DOE touts the partnership between the DOE, the facilities, and the users. However, when policy decisions involving users are often made it is without prior consultation and coordination. That is certainly true in the case of this policy and it has, and continues to be a problem for the facilities and for the teams involved. One of the less tangible issues involved in this is the role of university users. Many of the beamline teams that were funded were made of university users. This allowed them to bring their intellectual capital to the facilities and to participate as full members. Without this type of funding, the interaction of the university scientists, including post docs and students will be reduced. This, in turn, will reduce the intellectual involvement in the facility as well as the nature of the involvement of many graduate students and post docs. Unless they are from universities near the facilities, few will have the opportunity to spend the amount of time working with and on the beamlines to get the training needed for future synchrotron and beamline scientists. For those university groups who had been particularly successful, it is certainly not clear that some accommodation could be made. This is a consequence of

the DOE decision that is still playing. It's long term impact is still to be seen but in the short term this has left a very bitter taste in the mouths of many of the groups who have been caught in this transition. In the case of the X-11 PRT, the situation was even more troublesome. The renewal period when this decision was made occurred during a change of program monitors at DOE. With that change there was, at the very least, a breakdown of communication about the status of our project for renewal. When the renewal proposal was submitted in June 2000, we were told by our program officer that it was not appropriate since the previous year was a "termination" year. When we supplied the appropriate documentation to the contrary the proposal was allowed to go forward into review. However, no decision for funding was made by the usual time and we were told by our program officer that the matter was being reviewed but that if we pressed for a decision sooner rather than later we "might not like the answer." We were also encouraged to speak to the NSLS which we of course did, but the timing within their budget cycle made it impossible for them to come up with new money to support or even help underwrite our effort. At no time was there any leadership shown by DOE nor any coordination attempted by them to try to find an acceptable decision and transition. Finally, in December 2000, less than two weeks before the formal end of the current project, it was insisted by us that a decision be made since we would have had no other choice than to cease operations and terminate all employees by December 15. We were then told that we could expect a onetime amount of \$95,000 (as opposed to our usual \$280,000) but that this would have to be written as a new project which resulted in a 4 month gap in the funding.

Although no one will admit it publicly, it was clear that we had been effectively written out of the budget in error and rather than admit this and try to come up with a more satisfactory solution we were left dangling for several months and then given this belated 3 months of support. The toll on all of the core PRT members and employees was terribly high, trying to keep operations going in the face of sudden termination. Even worse, this treatment showed a callous disregard for the contributions that this team and many of its individuals had made to the entire synchrotron community and represented very questionable management practices on the part of those involved at DOE. This was demonstrated, in part, by the lack of understanding of the impact a significant gap in funding. It makes one wonder how many of the program officers have had any experience in running their own competitive research efforts that involve supporting people who expect their salaries to be paid on a regular basis. This is not the only example I have seen within the entire synchrotron community of the relative weakness in providing leadership and oversight of both the facility and research operations. While there is a long history of successful construction project management, the record is much more spotty when it comes to operation.

In summary, while none of this can be reversed, at the very least, one hopes that some critical feedback will be accepted and understood by the relevant DOE staff involved. The public position of support and partnership needs to be consistent with the internal understanding of the contribution and value to the overall community as demonstrated through the funding and policy decisions affecting them. To be publicly praised but privately treated as part of a bean counting exercise without standard human values involved does not reflect well on the agency.

APPENDIX

PUBLICATION LIST FOR BEAMLINE X11-A

1999 – Present

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