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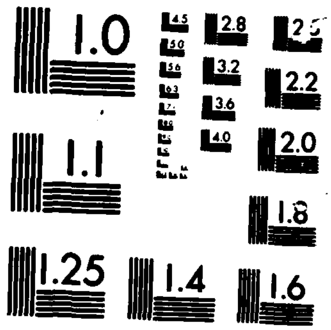

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SEMIANNUAL REPORT

January 1, 1985 to June 30, 1985

SUPERCONDUCTING ELECTRONIC FILM STRUCTURES

By

A. I. Braginski and J. R. Gavaler

Westinghouse Electric Corporation

Research and Development Center

Pittsburgh, Pennsylvania 15235

AFOSR Contract No. F49620-85-C-0043

Research sponsored by the Air Force

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Data from Nb/Sn and Nb/Al diffusion couple experiments provided supporting evidence for a proposed hypothesis that superconducting A15 compounds are formed via an oxygen catalyzed reaction. Both epitaxy, and the addition of an impurity (carbon) were successful in increasing the critical temperatures of NbN deposited at low temperature. Critical temperatures of over 16K were obtained in epitaxially grown NbN films sputtered on substrates held at less than 100°C. RHEED and X-ray rocking curve data show that the new UHV deposition and analytical facility has the capability for epitaxially growing high quality single crystals of the technologically important A15 and B1 superconductors. Low-leakage all-NbN tunnel junctions have been developed with ion-beam oxidized Al and Mg barriers, or rf-sputtered MgO barriers. The first Nb-Sn based junctions with refractory counterelectrodes were fabricated. XPS, RHEED, and tunneling have been used to characterize: 1) the structure of epitaxial films, 2) the role of ion-beam oxidation in the preparation of tunnel

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barriers that can be used with refractory counterelectrodes, and 3) anisotropic surface oxide growth on single-crystal films. Nb single crystal films were prepared which have three times lower rf surface losses compared to polycrystalline Nb.

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1. Semiannual Report, Superconducting Electronic Film Structures

January 1, 1985 to June 30, 1985

AFOSR Contract No. F49620-85-C0043

A. I. Braginski and J. R. Gavaler

2. ABSTRACT

Data from Nb/Sn and Nb/Al diffusion couple experiments provided supporting evidence for a proposed hypothesis that superconducting A15 compounds are formed via an oxygen catalyzed reaction. Both epitaxy, and the addition of an impurity (carbon) were successful in increasing the critical temperatures of NbN deposited at low temperature. Critical temperatures of over 16K were obtained in epitaxially grown NbN films sputtered on substrates held at less than 100°C. RHEED and X-ray rocking curve data show that the new UHV deposition and analytical facility has the capability for epitaxially growing high quality single crystals of the technologically important A15 and B1 superconductors. Low-leakage all-NbN tunnel junctions have been developed with ion-beam oxidized Al and Mg barriers, or rf-sputtered MgO barriers. The first Nb-Sn based junctions with refractory counterelectrodes were fabricated. XPS, RHEED, and tunneling have been used to characterize: 1) the structure of epitaxial films, 2) the role of ion-beam oxidation in the preparation of tunnel barriers that can be used with refractory counterelectrodes, and 3) anisotropic surface oxide growth on single-crystal films. Nb single crystal films were prepared which have three times lower rf surface losses compared to polycrystalline Nb.

3. OBJECTIVES

The objectives of the Westinghouse-AFOSR Program are:

1. Investigate the low-temperature synthesis of high-critical-temperature superconducting films.
2. Grow epitaxially single-crystal superconducting films and coherent layered structures.
3. Characterize the near-boundary crystalline and phase perfection of superconducting layer surfaces and interfaces, mostly by *in-situ* methods.
4. Study tunneling into high-critical-temperature (T_c) superconducting films.
5. Study radio-frequency surface losses in high-T_c superconducting films.
6. Investigate artificial tunnel barriers.

4. ACCOMPLISHMENTS

4.1 Preamble

This five-year research program was initiated in January 1983. It is aimed at understanding and improving the superconducting and normal state properties of layered, epitaxial, thin film structures incorporating high-critical-temperature superconductors. Anticipated results are intended to form a material science base for a future technology of high operating temperature superconducting electronics. The initial work in this program was performed under a contract covering the period from January 1, 1983 to December 31, 1984. In the first half of 1985 the level of effort was augmented to include an additional Objective (No. 6). Insulating tunnel barriers investigated under this task will include aluminum and other metal oxides. Understanding of the barrier physics is essential in order to develop to the fullest the implications of the entire research effort for the Air Force's technological needs in superconducting circuitry. Studies performed during the period from January 1, 1985 to June 30, 1985 are described in this report.

4.2 Low-Temperature Synthesis of High T_c Films

Low-temperature synthesis of high T_c superconducting films is required for S-I-S tunnel junction fabrication to avoid barrier damage. It is also of considerable scientific interest to further the understanding of stable and metastable compound formation. Progress on studies of both A15 and B1 structure materials have been made and are discussed below.

4.2.1 A15 Structure Compounds

Work has continued on the study of impurity-influenced low-temperature diffusion reactions between A and B elements to form

A15 structure, A_3B compounds. This investigation involved the formation of Nb-Sn and Nb-Al couples (bi-layers). Ultra-pure Nb and Sn or Al layers were sequentially deposited on sapphire or Nb substrates and then were annealed at various temperatures for various periods of time. The most significant result from the Nb-Sn study is the direct evidence that the elimination of grain boundaries in single crystal couples inhibits the formation of A15 phase at lower temperatures (650°C) and in the absence of grain boundaries. In identical conditions, polycrystalline Nb and Sn layers reacted to form the A15 phase. The results show that grain boundaries play a direct role in the formation of the Nb_3Sn A15 phase and are not merely affecting the rate of A15 growth. These data were reported at the March 1985 American Physical Society and at the conference on "Materials and Mechanisms of Superconductivity".

To explain these results, it was proposed that the Nb-Sn A15 phase, and in fact all A15 phases, nucleate from (or in the presence of) oxide, in analogy to β-tungsten. This means that oxygen must be present in the system, and can thus be considered as a catalyst for the A15 growth. It was also suggested that grain boundaries provide an increased surface area for the oxides to form, thereby promoting A15 phase nucleation. A direct, definitive demonstration that the absence of oxygen prevents the formation of A15 Nb_3Sn phase could not be obtained. In spite of the use of ultrapure Nb and a processing in ultra-high-vacuum, $p < 5 \times 10^{-11}$ torr, minute amounts of oxygen were always present in single crystal couples. In contrast, a direct proof was obtained that in Nb-Al diffusion couples the presence of Al_2O_3 is necessary to form the A15 phase. Without Al_2O_3, the A15 does not form in polycrystalline couples, even upon annealing at 900°C. The preliminary results on the Nb_3Al formation will be presented in a communication, to be submitted to Appl. Phys. Letters, and at the 1985 CEC-ICMC.

The hypothesis that the formation of all the high-T_c A15 structure superconductors occurs via intermediate surface oxide is admittedly a very provocative one since it challenges assumptions contained in a vast body of literature, encompassing hundreds of scientific papers. The evidence obtained thus far in the program certainly does not prove the universality of the effect. It is sufficiently strong, however, to warrant further studies on this problem.

4.2.2 B1 Structure Compounds

At present, most of the work on the low-temperature growth of high T_c superconductors has focussed on the B1 structure compound NbN. In the A15 compounds, structural disorder has a very deleterious effect on T_c. For this reason high critical temperatures have not been obtained in A15 films deposited at temperatures much below 700°C. In NbN, structural disorder has only a minimal effect on critical temperature. Therefore it has been possible, as reported by various workers, to prepare NbN at or near room temperature with nearly optimum T_c's. In this program, conditions have been established for depositing NbN films by magnetron sputtering at temperatures of $\lesssim$ 300°C which have critical temperatures of $\gtrsim$ 15K. Despite the successes that have been achieved in this area, an understanding of why and how certain experimental variables promote the low-temperature formation of the high-T_c NbN phase is still lacking. In this program efforts to gain such an understanding has centered on determining the influence of impurities and epitaxy on achieving the high-T_c phase. NbN films were deposited on sapphire, silicon, and MgO substrates under the same experimental conditions. The deposition temperatures ranged from 20 to 300°C. Depending on substrate temperature the films on silicon and sapphire had T_c's from 9 to 12°K , while those on MgO had systematically higher T_c's from 12 to 15K. Similar increases in critical temperature were observed through the addition of carbon (CH_4) into the sputter gas. These results thus support the hypothesis that the superconducting cubic NbN phase, at the composition which has the highest T_c, is relatively less stable than the competing non-superconducting phase(s) having similar compositions. The highest-T_c NbN composition must therefore be stabilized either by an impurity (such as carbon) or by epitaxy.

The experimental results highlighted here will be presented at the 1985 CEC-ICMC.

4.3 Epitaxial Growth of Superconducting Films

The investigation of epitaxial growth processes has a technological as well as a scientific motivation. Elimination of near-surface structural disorder in layered film structures will make high-T_c S-I-S tunnel junctions possible. Epitaxy has been shown useful in stabilizing high-T_c Nb_3Ge and, as described in the preceding section, NbN. Finally single crystals of high-T_c superconductors will permit the investigation of their intrinsic properties and will advance the science of superconductivity.

As described previously, a new type of ultra-high vacuum deposition and in-situ analytical facility, referred to as the Superlattice Analytical and Deposition (SDAF) has been implemented for use in this program. Using this facility, it has been found that in many cases epitaxial single crystal film growth can be very easily achieved, providing that the substrates have good surface quality. This means that the main requirement for growing single crystal films is to prepare substrates which have clean and damage-free single crystal surfaces. This requirement is easily met in the case of sapphire and MgO substrates. Niobium, Nb_3Sn, Nb_3Ge, and Mo-Re single crystal films have been grown on sapphire. The evidence for single crystallinity is primarily from in-situ RHEED. X-ray rocking curve data has also been obtained for Nb (0.1° rocking curve width), Nb_3Sn (0.4°), and $Mo_{65}Re_{35}$ (0.3°). Single crystal NbN films have been epitaxially grown onto both sapphire and MgO substrates.

The problem of surface preparation was found to be somewhat more severe in the case of Nb_3Ir which was chosen as the ideal substrate material for the deposition of Nb_3Ge. This problem has however been resolved and single crystal Nb_3Ge has now been grown on Nb_3Ir. The quality of these single crystal and the superconducting and tunneling properties of the Nb_3Ge overlayers have not as yet been assessed. More complete discussions of all the epitaxial film work completed thus far in this program are contained in papers presented at the "Materials and Mechanisms Conference, the "IC SQUID 85" Conference and the ICMC. In general, the capability that has been developed to grow single crystal films of the A15 and B1 superconductors is perhaps the most significant achievement of this program thus far. One of the main obstacles preventing a more complete understanding of high-T_c superconductors has been the paucity of good single crystals. It would appear that this obstacle can now be removed.

4.4 Characterization of Near-Surface Layers

The purpose of this task is to develop and apply methods of surface and interface characterization that are appropriate for the *in-situ* investigation of thin films and layered structures generated under other tasks of the program. The role of near-surface characterization is somewhat different for each task. However, crystallinity, phase composition, and physical uniformity are of interest for all surfaces. Most films were deposited on single-crystal sapphire, silicon, or MgO substrates. Reflection High-Energy Electron Diffraction (RHEED), which probes approximately 50 Å into a smooth surface, has been the primary technique used for identifying epitaxial relationships between substrates and films. The azimuthal angles at which low-index electron diffraction patterns can be observed, are routinely recorded for substrates and films. As an example, single-crystal NbN films with a (111) growth direction were deposited on (0001) sapphire. The RHEED patterns showed that the (110) direction in the plane of the film was parallel to the $(10\bar{1}0)$ direction in the sapphire. For thick films, the epitaxial relationship has been confirmed by X-ray diffraction, but RHEED has been essential for studying the crystal structure of either the initial growth of a thick film or very thin films such as tunnel barriers.

Tunneling was used to probe the top 50 Å of homo-epitaxially grown NbN films. The T_c of the top 1000 Å layer deposited at 100°C cannot be measured independently of the T_c of the bottom NbN layer grown at 700°C. However, tunneling measurements of the energy gap of the top layer established that the T_c of the low-temperature film was > 16K — about 3K higher than films grown at 100°C on other substrates. The near-surface characterization of tunnel barriers and barrier/electrode interfaces in tunnel junctions is included in sections 4.5 and 4.7.

4.5 Tunneling Into High-T_c Superconductors

Several types of low-leakage tunnel junctions have been formed with NbN counterelectrodes. The parameters of barrier formation have been most carefully studied for ion-beam oxidized barriers. It has been found that the ion-beam energy must be 300 ± 50 V to obtain low-leakage junctions. Typical exposure to the ion beam was 20 A-min/cm^2. Based on a typical i_cR_n product of 2 mV, $V_m \approx$ 15 mV was measured at 2.5 mV. The curvature of the junction I-V curves in the range of 0.1-0.4V was used to infer barrier widths and heights for comparison with thermal oxide barriers whose thicknesses were measured by XPS, and with other barrier materials.

NbN counterelectrodes have also been deposited on Nb_3Sn/ion-beam-oxidized Al bilayers. These are the first Nb_3Sn-based tunnel junctions with refractory counterelectrodes. The V_m was approximately 5. A comparison of junctions with thermal oxide ($V_m \sim$ 80mV) and ion-beam oxide barriers, both with Pb-Bi counterelectrodes, showed that the ion beam (300 V) damaged the surface layer of the Nb_3Sn.

4.6 Radio-Frequency Surface Losses in High-T_c Superconducting Films

The measurements of single-crystal niobium films supplied to MIT - Lincoln Laboratory (LL) resulted in resonator Q-values up to 10^5 at 3-4 GHz and T $\approx$ 5K. This represents a reduction in rf-surface loss by a factor of at least 3, compared with polycrystalline Nb. The frequency dependence of losses (Q) was in agreement with theory. Additional single crystal Nb films were prepared on (0001) sapphire to test the effect of crystal orientation on rf losses. These samples were shipped to LL. Upon completion of Nb film measurements at LL, single crystal NbN films will be fabricated for rf loss measurements.

4.7 Artificial Tunnel Barriers

Ion-beam-oxidized Al and Mg metallic overlayers on NbN were studied to obtain low-leakage tunnel junctions with NbN counterelectrodes. The ion beam treatment in an argon-oxygen atmosphere removed the surface oxide while producing a thicker oxide than was initially formed by room-temperature thermal oxidation. X-ray Photoelectron Spectroscopy (XPS) was used to measure the oxide thickness and to determine the process end-point before the ion beam oxidized the top surface of the base superconductor. RHEED measurements showed that the oxide barriers were randomly-oriented polycrystalline after thermal oxidation, and still crystalline after ion-beam oxidation, but with more diffuse rings in the diffraction pattern.

XPS measurements of oxidized Al and Mg overlayers on NbN have been made as a function of sample temperature up to 800°C. In contrast to the slow decrease of unoxidized Al thickness as a function of analysis temperature, the unoxidized Mg started to diffuse above 300°C and completely disappeared above 500°C. There was no change in the thickness or chemical shift of the Al or Mg oxide. On this basis, processing temperatures (counterelectrode deposition) up to 800°C appear to be feasible. However some degradation of the NbN base electrode energy gap would occur above 300°C in the case of oxidized Mg barriers.

Oxidized metal overlayers have been used for oxide barrier formation because it was thought that a thin metallic layer would cover another metal more uniformly than would an oxide deposited directly. The direct deposition of oxide barriers by rf sputtering is now also being pursued in parallel, as a possible route to all-epitaxial NbN/MgO/NbN tunnel junctions. All NbN junctions have been fabricated with composite barriers of Nb_2O_5 (native oxide) and rf sputtered MgO. The composite barriers resulted in junctions with a lower V_m at 2.5 mV than the ion-beam oxidized barriers, $V_m \approx 10$ mV. One issue to be addressed in producing an epitaxial multilayer by this method is that the surface of the NbN base electrode starts to oxidize as soon as the sputtering of MgO begins.

Alternative insulators for tunnel barriers have also been tested. Of these, calcium fluoride has worked best, but only for NbN base electrodes which already had a low-resistance native-oxide barrier to seal pinholes in the CaF_2. The lattice constant of CaF_2 closely matches that of Nb_3Sn and may, therefore, be a good base for the growth of a Nb_3Sn counterelectrode.

5. PUBLICATIONS

1. "Reactively Sputtered V_3Si and Nb_3Ge Films,"
   J. R. Gavaler and J. Greggi IEEE Trans on Magn. vol. MAG. 21, 417, (1985).

2. "Tunnelling and Interface Structure of Oxidized Metal Barriers on A15 Superconductors,"
   J. Talvacchio, A. I. Braginski, M. A. Janocko, and S. J. Bending; IEEE Trans. on Mag. Vol. MAG-21, 521 (1985).

3. "Epitaxial Growth of High-T_c Superconducting Films
   J. R. Gavaler, A. I. Braginski, M. A. Janocko, and J. Talvacchio
   Materials and Mechanisms of Superconductivity, to be published.

4. "Detection of Bound Vortex-Antivortex Pairs in Superconducting Thin Film by Surface Acoustic Waves,"
   A Schenstrom, M. Levy, H. P. Fredricksen, and J. R. Gavaler;
   Materials and Mechanisms of Superconductivity, to be published.

5. "Artificial Oxide Barriers for NbN Tunnel Junctions,"
   J. Talvacchio, J. R. Gavaler, A. I. Braginski, and M. A. Janocko;
   Submitted to the J. Appl. Phys.

6. "A LEED, AES, and XPS Study of Single Crystal Nb_3 Ir Surfaces,"
   S. Sinharoy, A. I. Braginski, J. Talvacchio, and E. Walker;
   Submitted to J. Surface Science.

7. "New Materials for Refractory Tunnel Junctions: Fundamental Aspects,"
   A. I. Braginski, J. R. Gavaler, M. A. Janocko, and J. Talvacchio;
   Will be submitted for publication in the proceeding of IC SQUID.

8. "Epitaxial Growth of NbN,"
   J. R. Gavaler, J. Talvacchio, and A. I. Braginski;
   Will be submitted for publication in the proceeding of ICMC.

9. "Formation of A15 Phase in Epitaxial and Polycrystalline Nb-Sn and Nb-Al Diffusion Couples,"
A. I. Braginski, and J. R. Gavaler;
Will be submitted for publication in the proceeding of ICMC.

10. "UHV Deposition and In-Situ Analysis of Thin-Film Superconductors,"
J. Talvacchio, M. A. Janocko, J. R. Gavaler, and A. I. Braginski;
Will be submitted for publication in the proceeding of ICMC.

6. PERSONNEL

A. I. Braginski J. R. Gavaler	Principal Co-Investigators
J. Greggi	
M. A. Janocko	
S. Sinharoy	
J. Talvacchio	

7. COUPLING ACTIVITIES *

1. "Tunneling and Properties of Superconducting Mo-Re Films," J. Talvacchio, M. A. Janocko, and A. I. Braginski; Contributed talk at the March, 1985 A.P.S. Meeting.

2. "A LEED Auger and XPS Study of Single Crystal Nb_3Ir Surfaces," S. Sinharoy, A. I. Braginski, and E. Walker; Contributed talk at the March, 1985 A.P.S. Meeting.

3. "The Effect of Oxygen and Grain Boundaries on the Formation of the A15 Phase in Nb-Sn Diffusion Couples," J. R. Gavaler and A. I. Braginski; Contributed talk at the March, 1985 A.P.S. Meeting.

4. "Microchemical Analysis of High-T_c A15 Structure Films," J. Greggi and J. R. Gavaler; Contributed talk at the March, 1985 A.P.S. Meeting.

5. "Deposition and Analysis of High-T_c Superconducting Films," J. Talvacchio; Seminar at Stanford University, April 1985.

6. "Ultra-High-Vacuum (UHV) Closed System for Fabrication and In-Situ Analysis of Metallic Thin Film Structures," A. I. Braginski; Seminar at the University of Wisconsin, Madison. April, 1985.

7. "Epitaxial Growth of High-T Superconducting Films," J. R. Gavaler, A. I. Braginski, M. A. Janocko, and J. Talvacchio; Invited talk at the "Materials and Mechanisms of Superconductivity" Conference Ames, Iowa (May, 1985).

8. "New Materials for Refractory Tunnel Junctions - Fundamental Aspects," A. I. Braginski, J. R. Gavaler, M. A. Janocko, and J. Talvacchio; Invited talk at IC SQUID, Berlin, F.R.G. June, 1985.

* Speaker's Name is Underlined.

9. "High-T_c Superconducting Thin Film Structures for Advanced Electronic Applications,"
R. D. Blaugher;
Invited talk at the U.S.-Japan Workshop on Josephson Junction Electronics. Honolulu, Hawaii (June, 1985).

10. "The Use of an UHV Deposition and Analytical Facility for Studies of Metallic Thin Film Structures,"
A. I. Braginski;
Seminar at KfK, Karlsruhe, F.R.G. (July, 1985).

8. PATENTS AND INVENTIONS

None

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