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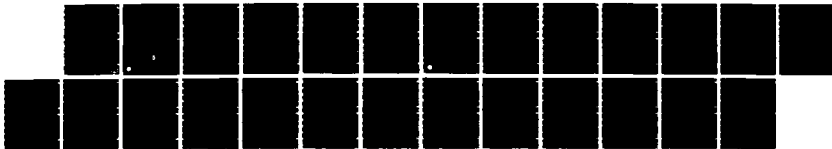
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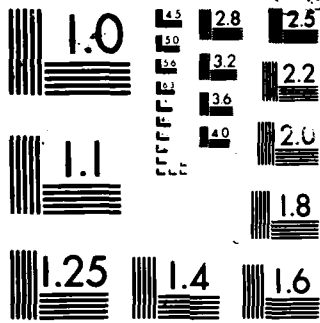
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SUPERCONDUCTING ELECTRONIC

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FILM STRUCTURES

By

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A. I. Braginski and J. R. Gavaler

Westinghouse Electric Corporation
Research and Development Center
Pittsburgh, Pennsylvania 15235

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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) junctions processed at temperatures up to 800°C. Nb single crystal films were prepared which have three times lower rf surface losses compared to polycrystalline Nb.

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1. Annual Report, Superconducting Electronic Film Structures.

January 1, 1985 to December 31, 1985

AFOSR Contract No. F49620-85-C-0043

A. I. Braginski and J. R. Gavaler.

ANNUAL REPORT

January 1, 1985 to December 31, 1985

SUPERCONDUCTING ELECTRONIC
FILM STRUCTURES

By

A. I. Braginski and J. R. Gavaler

Westinghouse Electric Corporation
Research and Development Center
Pittsburgh, Pennsylvania 15235

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2. ABSTRACT

Results from Nb/Sn and Nb/Al diffusion couple experiments show that oxygen or oxides can have a positive influence on the nucleation and growth of stable as well as metastable A15 phases. Data on the effect of epitaxy and the addition of carbon impurities on the critical temperatures of NbN films deposited at low temperatures show that both are effective in stabilizing the stoichiometric B1 phase. Critical temperatures of 16.4K were obtained in epitaxially grown NbN films deposited on $< 100^\circ\text{C}$ substrates. According to RHEED and X-ray rocking curve data, high quality single crystal films of the technologically important A15 and B1 superconductors, including Nb-Sn, Nb-Ge, and NbN can be reproducibly grown in the new deposition and analytical facility. 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) junctions processed at temperatures up to 800°C . 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 1985 the level of effort was augmented to include an additional Objective (No. 6). Insulating tunnel barriers investigated under this task 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 December 31, 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 at reasonably low temperatures. Progress on studies of both A15 and B1 structure materials is 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 the A15 phase at lower temperatures (650°C). In the absence of grain boundaries, both electrical and X-ray measurements indicated the presence of only the non-superconducting Nb₆Sn₅ phase. Under 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 Nb₃Sn A15 phase and are not merely affecting the rate of A15 growth. A complete version of these data was presented at the CEC-ICMC conference and is included in the Proceedings.

In this paper, it was proposed that the Nb-Sn A15 phase, and perhaps all A15 superconductors, nucleate from (or in the presence of an oxide) in analogy to how the prototype A15 material, β -tungsten, is formed. It was suggested that in the Nb-Sn bilayers, grain boundaries promoted A15 phase nucleation by providing an increased surface area for the oxides to form. The concept that all high-T_c A15 structure superconductors are formed via an intermediate surface oxide is admittedly a very provocative *once*, since it challenges assumptions contained in a vast body of literature. A definitive validation of this concept would require the demonstration that elements such as Nb and Sn, which under ordinary conditions easily form an A15 phase, do not do so in the total absence of oxygen impurities regardless of temperature. An attempt to provide such a demonstration was made by reacting ultra-pure niobium and tin under ultra-high purity conditions at 850°C. Despite the very low level of oxygen present under these conditions, the A15 phase was still formed. However, evidence supporting the importance of oxygen or oxides in the growth of the A15 phase was obtained in the Nb-Al system. At annealing temperatures of 850°C, Nb-Al bilayers deposited on oxide substrates reacted to form the A15 phase. Similar layers deposited on Nb substrates did not. However at higher temperatures the A15 phase was obtained in all cases. Data on the Nb/Al diffusion experiments will be reported at the March 1986 American Physical Society Meeting.

In essence, results obtained thus far on the effect of oxygen or oxides on the formation of the A15 phase are somewhat contradictory. The supporting evidence is sufficiently strong, however, to warrant further studies of this problem.

4.2.2 B1 Structure Compounds

During the past year, most of the work on the very low temperature (i.e. close to room temperature) growth of high- T_c superconductors has been focussed on the B1 structure compound NbN. In this program, it has previously been shown that A15 structure V_3Si having ideal 3/1 stoichiometry can be grown at temperatures of less than 300°C. However, because of structural disorder, the T_c of this material is only ~ 7K. In NbN, structural disorder has only a minimal effect on critical temperature. Therefore it has been possible, as reported by workers in various laboratories, to prepare NbN at or near room temperature with nearly optimum T_c 's of ~ 16K. Despite the successes that have been achieved in growing high- T_c NbN at low temperatures, an understanding of the growth conditions which allows this compound to form at such temperatures has been lacking. Indeed in many cases, workers reporting the successful growth of high- T_c NbN films have tended to ignore the well-documented fact that the superconducting B1 structure NbN phase is unstable below ~ 1300°C and therefore should not have formed under the conditions reported. In this program, efforts to gain an understanding of NbN growth at low temperatures has centered on determining the influence of impurities and epitaxy on stabilizing the B1 phase.

Initially, NbN was deposited under as high purity conditions as possible. Under these high purity conditions, the superconducting B1 phase was found to still form, however with a depressed T_c of < 10K. The formation of this low- T_c B1 phase was attributed to the stabilizing effect, on the B1 structure, of the residual gas impurities in the system. Analysis of the residual gases in the deposition system indicated that the prime impurity was methane (CH_4). It is known from the literature that carbon which would enter the NbN via the methane gas, does in fact stabilize the B1 phase at low temperatures. In the present deposition system, the amount of methane was not sufficiently high to allow the formation of the stoichiometric single-phase

B1 material, which would have a higher T_c . Optimum T_c 's of 15 to 16K were achieved by the deliberate addition of more impurity (methane) into the sputtering gas.

Stabilization of the high- T_c B1 phase was also achieved by epitaxially growing the NbN films on B1 structure substrates without the addition of more impurities. Improved T_c 's were obtained in NbN films deposited on polycrystalline MgO, however the highest values were obtained in films deposited on single crystal MgO or NbN substrates. Homoepitaxy of NbN produced a stoichiometric single crystal compound with a T_c of $> 16K$ at a deposition temperature below $100^\circ C$. The single crystal substrates presented an exclusively B1 surface for nucleating the B1 phase. In polycrystalline material, preferential nucleation of the B1 phase would occur on the grains, however, the undesired lower energy non-superconducting NbN phase(s) could still nucleate at the grain boundaries.

In summary, the data indicate that the stabilization of the NbN B1 phase, by inhibiting the nucleation of non-superconducting phase(s), is required to obtain optimum T_c 's in NbN films deposited at low temperatures. This can be achieved either via epitaxy or by the addition of impurities into the NbN structure. A complete discussion of all of these data is contained in a paper presented at the 1985 CEC-ICMC and included in the Proceedings.

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 Nb₃Ge 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 (UHV) 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, Nb₃Sn, Nb₃Ge, 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), Nb₃Sn (0.4°), and Mo₆₅Re₃₅ (0.3°). Single crystal NbN films have been epitaxially grown onto both sapphire and MgO substrates. A review of results on superconducting film epitaxy is included in a paper presented at the 1985 "Materials and Mechanisms of Superconductivity" conference and included in the Proceedings.

The problem of surface preparation was found to be more severe in the case of Nb₃Ir which was chosen as the ideal substrate material for the deposition of Nb₃Ge in UHV. This problem has however been resolved and single crystal Nb₃Ge has now been grown on Nb₃Ir. The study of Nb₃Ir crystal surfaces, their cleaning and observed surface reconstructions was presented in a paper being published in "Surface Science." Single crystal Nb₃Ge films have been obtained on (100), (110) and (111) bulk single crystals of Nb₃Ir. Additionally, single crystal Nb₃Ge was grown on (100) single crystal Nb₃Sn films. According to RHEED, all deposits were single-crystalline with the orientation defined by the substrate. On Nb₃Ir the average composition determined by electron microprobe was close to 3:1 stoichiometry. X-ray diffraction using a cylindrical texture camera indicated a lattice parameter mismatch between the single crystal films and the Nb₃Ir substrates, and also the presence of a polycrystalline second phase. Inductive measurements of critical temperature, T_c indicated a low T_c of Nb-Ge, not exceeding 11K. Quasiparticle tunneling results for Nb-Ge on Nb₃Sn also indicated a low T_c. These low T_c's suggest that heteroepitaxy alone is not sufficient to insure the nucleation and growth of the high-T_c Nb₃Ge phase when a sufficient level of oxygen impurity is not present. However, deposition rate fluctuations during the Nb-Ge epitaxial growth were high enough to nucleate a more stable α -phase. Until these fluctuations are reduced, the question whether

stabilizing high- T_c Nb_3Ge can be achieved by epitaxy alone remains open. Data on the Nb-Ge epitaxy will be presented at the 1986 March APS Meeting.

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. This obstacle is now being removed. For example, in collaboration with the MIT, AFOSR program on superconductivity, the upper critical field in single crystal NbN is being investigated. Direct evidence of H_{c3} was obtained in (100) NbN single crystal films. Additional anisotropy in upper critical field was observed in (111) NbN.

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 (1010) 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. RHEED data have been published in most of the papers generated during the course of this program.

A reverse-view LEED apparatus was acquired and installed (at no cost to the AFOSR) in the third quarter of 1985. The low energy of the incident electrons permitted the characterization of structure in the top monolayer of a film. Surface reconstructions were observed for certain orientations of single-crystal, high- T_c films: (1 x 2) reconstruction on Nb_3Sn (100), and (3 x 3) reconstruction on NbN (111). The epitaxial growth of Nb films was observed with LEED for films as thin as 5 Å. Films deposited on sapphire substrates continued to grow as single crystals. However, Nb deposited on Si (100) substrates appeared to grow epitaxially only up to a thickness of 5 Å. At a thickness of 40 Å, a fine-grained, polycrystalline film was observed. The work on Nb films was performed in collaboration with the Stanford University AFOSR program in superconductivity.

Tunneling was used to detect superconductivity in the top 50 Å of films deposited on high- T_c underlayers. Examples are the homo-epitaxial growth of NbN below 100°C on a single-crystal underlayer formed at 700°C, and the hetero-epitaxial growth of Nb_3Ge on single-crystal Nb_3Sn . In the first case, tunneling measurements on the energy gap of the top NbN 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 the ion-beam oxidized barriers. It has been found that the ion beam energy must be $300 \pm 50V$ to obtain low-leakage junctions. Typical exposure to the ion beam was $20 \mu A\text{-min}/cm^2$. Based on a typical $i_c R_n$ product of 2 mV, $V_m \approx 15$ mV measured at 2.5 mV. The curvature of the junction I-V curves in the range of 0.1 to 0.4V was used to infer barrier widths and heights for comparison with thermal oxide barriers, barrier thicknesses measured by XPS, and other barrier materials. These data will be published in the Proceedings of the Third International Conference of Superconducting Quantum Devices.

NbN counterelectrodes have also been deposited on Nb₃Sn/ion-beam oxidized Al bilayers. These are the first Nb₃Sn-based tunnel junctions with refractory counterelectrodes. The V_m was approximately 5. A comparison of junctions with thermal oxide ($V_m \approx 80$ mV) and ion-beam oxide barriers, both with PB-Bi counterelectrodes, showed that the ion beam (300V) damaged the surface layer of the Nb₃Sn. These results were presented at the 1985 ICMC, and will be published in the Proceedings.

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 to 4 GHz and $T \approx 5$ K. 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 and Nb₃Sn films will be fabricated for rf loss measurements.

4.7 Artificial Tunnel Barriers

Two divergent approaches have been used in an attempt to obtain low-leakage barriers by improving the uniformity of the barrier. The first approach involved an effort to form amorphous or very fine-grained crystalline barriers. The second was to grow a crystalline barrier epitaxially, and form a coherent structure with both electrodes.

Ion-beam oxidized Al or Mg metallic overlayers on NbN were studied as part of the first approach. The ion beam treatment in an argon-oxygen atmosphere removed the surface oxide while forming a thicker oxide than can be 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. NbN counterelectrodes were successfully deposited at a temperature of 300°C. The fabrication of artificial oxide barriers by ion-beam oxidation was published in the Journal of Applied Physics.

Co-sputtered Al-Mg overlayers on NbN were oxidized to form an amorphous barrier. Tunnel junctions completed by depositing NbN counterelectrodes had lower leakage currents than comparable junctions made with polycrystalline oxidized Al or oxidized Mg. A paper on properties of crystalline vs amorphous oxide barriers was prepared and is to be submitted to the Journal of Applied Physics.

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. This material was presented at the 1985 ICMC and will be published in the Proceedings.

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. However, the direct deposition of oxide barriers by rf sputtering or by evaporation of oxide sources are the most straightforward routes to all-epitaxial tunnel junctions. Epitaxial trilayers of NbN/MgO/NbN and NbN/Al₂O₃/NbN have been formed. Preliminary tunneling measurements of the NbN/MgO/NbN trilayers showed that the barrier did not cover completely. The deposition parameters that influence surface diffusion rates, such as substrate temperature and deposition rate, have yet to be optimized. Deposition temperatures for the barrier up to 700°C have been used without interdiffusion of the base and barrier materials. Nevertheless, voltage gap values exceeding 5 mV were obtained from the NbN/MgO/NbN trilayers. These results on the formation and

tunneling properties of these epitaxial barriers will be presented at the 1986 APS March Meeting.

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6. PERSONNEL

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7. COUPLING ACTIVITIES*

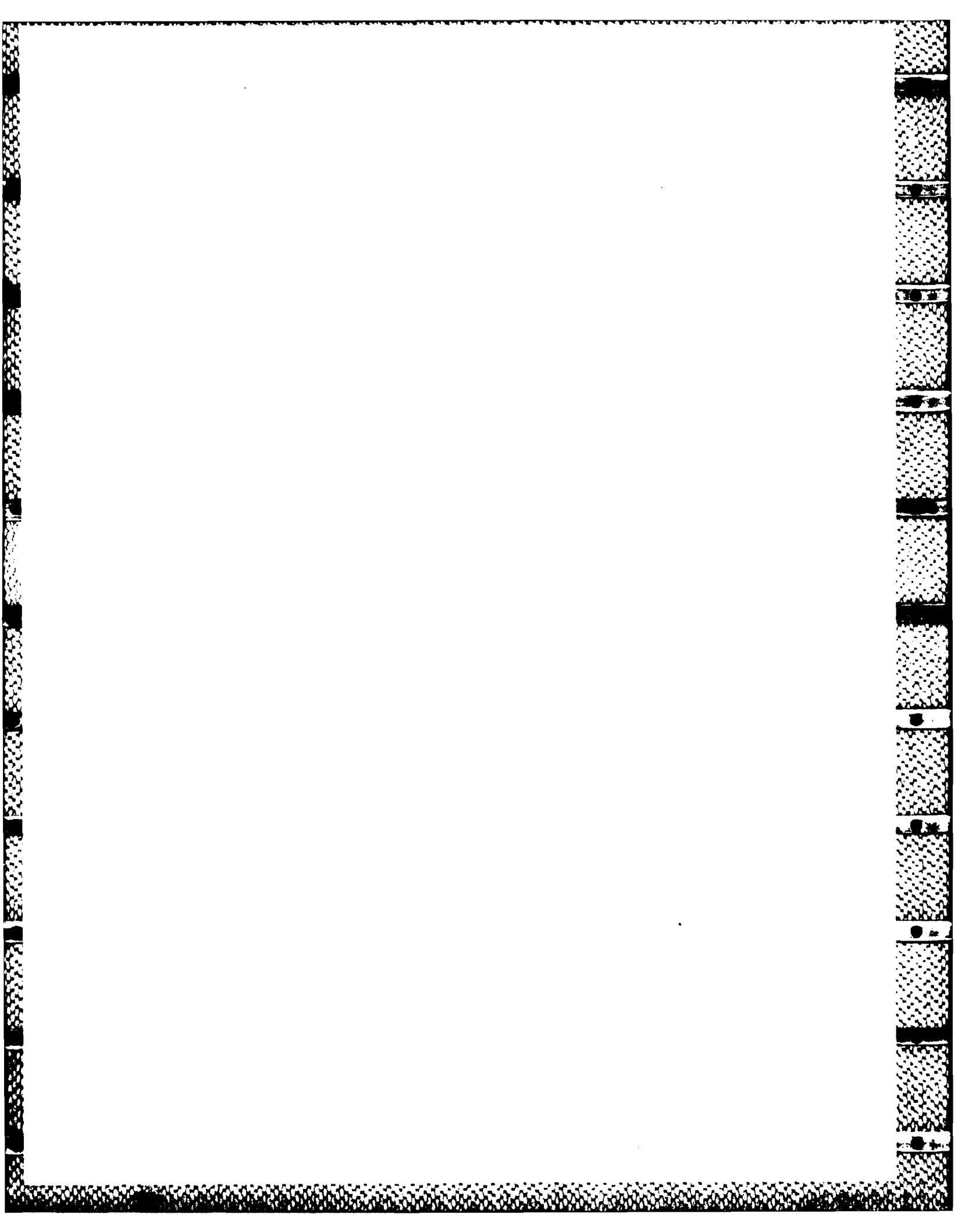
1. "Tunneling and Properties of Superconducting Mo-Re Films," J. Talvacchio, M. A. Janocko, and A. I. Braginski, Contributed talk at the March, 1985 A.P.S. Meeting.
2. "A LEED Auger and XPS Study of Single Crystal Nb₃Ir Surfaces," S. Sinharoy, A. I. Braginski, and E. Walker, Contributed talk at the March, 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. Gregg 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_c 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.
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).

*Speaker's name is underlined.

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).
11. "Epitaxial Growth of NbN," J. R. Gavaler, J. Talvacchio, and A. I. Braginski, Contributed talk at ICMC, Cambridge, MA (August, 1985).
12. "Formation of A15 phase in Epitaxial and Polycrystalline Nb-Sn Diffusion Couples," A. I. Braginski and J. R. Gavaler, Contributed talk at ICMC, Cambridge, MA (August, 1985).
13. "UHV Deposition and In-Situ Analysis of Thin Film Superconductors," J. Talvacchio, M. A. Janocko, J. R. Gavaler, and A. I. Braginski, Contributed talk at ICMC, Cambridge, MA (August, 1985).
14. "In-Situ Fabrication and Analysis of High- T_c Device Film Structures," A. I. Braginski, Invited talk at the Workshop on Superconductive Electronic Devices, Circuits and Systems, Waterville, NH, August 12, 1985.
15. "Niobium Nitride Junction Fabrication: An Example of In-Situ Approach," J. Talvacchio, Invited talk at the Workshop on Superconductive Electronic Devices, Circuits and Systems, Waterville, NH, August 12, 1985.
16. "Preparation and Growth of Superconducting Films," A. I. Braginski, Invited lecture at the Gordon Conference on Superconducting Films, Holderness, NH, August 26, 1985.
17. "Preparation and Growth of Superconducting Film Structures," A. I. Braginski, Seminar at the Electrotechnical Laboratory (MITI) Tsukuba-City, Japan, September 28, 1985.
18. "Preparation of Superconducting Film Structures in a UHV Systems," A. I. Braginski, Invited Seminar at MIT, December 9, 1985.

8. PATENTS AND INVENTIONS

None.



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