MATERIALS RESEARCH FOR HYDROGEN-COOLED
SUPERCONDUCTING POWER-TRANSMISSION LINES

Sixth Quarterly Report

Covering the Period

April 1, 1981 to June 30, 1981

A. M. Sletten, A. I. Braginski, M. Rosado,
J. R. Gavalier, J. Gregg, and J. Schreurs (Westinghouse)
and
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MASTER

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Westinghouse Electric Corporation

Research and Development Center

Pittsburgh, Pennsylvania 15235

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SUMMARY

The objectives of this three-year program are to:

- I. Perform dielectric breakdown and loss measurements in liquid hydrogen and liquid-hydrogen-impregnated synthetic dielectrics, at temperatures between 14 and 20°K and at hydrostatic pressures up to 5 atmospheres. Effects of dissolved impurities/additives in the liquid will be included in this study.
- II. Characterize the self-field and low-field superconducting properties of high-critical temperature materials at temperatures between approximately 14 and 20°K.

During the current reporting period, the following was accomplished:

- I.1 The construction of the dielectric test apparatus continued and the system of small electrodes was completed. Difficulties were encountered in the construction of the LH₂ vessels and the junction

box, which have retarded their completion. Other work to be performed at the Westinghouse R&D Center on the dielectrics part of this project has been postponed indefinitely due to the shortage of funding.

- II.1 Analytical work confirmed the formation of a V-Si-C pseudobinary compound with $T_c \approx 17K$. Observed Zr-contamination requires, however, additional investigation.
- II.2 Multiple Nb-Ge-Al specimens have been prepared and their T_c characterized.
- II.3 Additional results on laser annealing of Nb-Ge produced contradictory results and led to an intensive electron microscopic and analytical investigation.
- II.4 An apparatus for E-beam pulse annealing was assembled and is being tested.
- II.5 Prior fabrication capability was restored in the CVD system for Nb_3Ge fabrication.

1. R&D STATUS REPORT

1.1 Liquid Hydrogen Dielectric Study

1.1.1 Literature Search

Completed.

1.1.2 Design and Construction of Test Equipment

The system of small electrodes was built as shown in Figure 1. It consists of 20 concentric pairs of electrodes distributed on two levels. All pairs have adjusted gaps. Ten pairs are plane-to-plane and the other ten sphere-to-sphere. This large number of electrodes is used to study several gaps at a time, thus minimizing the times that the cryogenic system has to be cycled between ambient and LH₂ temperatures. It is also convenient in testing solid insulation, since it is assumed that each electrical breakdown will permanently damage the film of insulating solid between electrodes. Since only one valuable breakdown can be obtained per solid film, a large number of electrodes is needed to expedite data acquisition and minimize costs.

The construction of the vessels for LH₂ was hindered by difficulties in passing the X-ray inspection for the welded joints. The subcontractor building the vessels had to subcontract this part to another company to accomplish the requirements for ASME certification of the vessels.

The junction box leaked through the ceramaseal electrical feedthroughs when tested. The feedthroughs are suspected of being defective and efforts are being made to obtain new ones (at no cost) from the manufacturer.

The vessels and the junction box should be finished during the next quarter.

Figure 1 System of small electrodes.

Other work related to the dielectric properties of LH_2 has been stopped due to the insufficiency of allocated funds (effective April 1, 1981).

1.2 Superconducting Materials

1.2.1 Critical Temperature of Carbon-Doped V_3Si (Westinghouse)

1.2.1.1 Objective

The objective of this task and the background were described in the Second Quarterly Report.

1.2.1.2 Progress

Results which were discussed in detail in the Fifth Quarterly Report showed that the critical temperature of highly Si-deficient, low- T_c V-Si film can be enhanced by the implantation of carbon and subsequent annealing. This enhancement was limited, however, to a maximum value of $\sim 17\text{K}$ — the T_c of stoichiometric V_3Si . No enhancement was observed in films which were closer to stoichiometry and thus already had T_c 's near 17K . A T_c onset of 20.8K , reported in the literature⁽¹⁾ for a material having a composition $\text{V}_{73.85}\text{Si}_{24.23}\text{C}_{1.92}$ was not reproduced despite the fact that the experiments were designed so that at least some portion of one or more of the C-implanted films would have this composition. Based on T_c measurements, the composition of the films were deduced to be between $\text{V}_{81}\text{Si}_{19}$ and $\text{V}_{73}\text{Si}_{27}$ before implantation which spans the compositional range of interest. To insure the validity of these compositional data, several films were analyzed by Energy Dispersive X-ray Analysis (EDX). The analyses confirmed that the data were indeed valid. For example, a film assumed to be at or near the stoichiometric V_3Si composition based on its T_c of $\sim 17\text{K}$ was found to have the composition $\text{V}_{74.5}\text{Si}_{25.5}$. Another film which, from its T_c of $\sim 6\text{K}$ was believed to be highly Si-deficient, had a composition $\text{V}_{89.3}\text{Si}_{10.7}$ which is in good agreement with the value deduced from T_c . In substance the EDX data showed that any deficiencies in T_c 's in the V-Si-C films are not due to unexpected and thus erroneous V-Si compositions. What was unexpected, however, was the discovery that the annealed V-Si-C film contained

significant levels of zirconium. Since none of the unannealed samples contained this element it apparently was introduced during the annealing process. To determine what effect, if any, the Zr had on the T_c 's of the films another series of V-Si films will be C-implanted. These samples will then be annealed with appropriate precautions taken that no foreign elements such as Zr are introduced.

1.2.2 Phase Uniformity of Nb₃Ge (Stanford University)

1.2.2.1 Objective

The objective of this task is to investigate methods for improving the phase uniformity of Nb₃Ge in order to increase the T_c^* and the self-field J_c in the temperature range of liquid hydrogen. As discussed in the 1980 Annual Report the T_c^* of Nb₃Ge is significantly lower than its T_c , probably due to the A15 phase inhomogeneity. One of the approaches to improve the homogeneity is to substitute aluminum and form a pseudobinary compound Nb₃(Al, Ge). This A15 phase is more stable than Nb₃Ge and still has a T_c in excess of 20K so that a T_c^* approaching 20K could possibly be obtained.

1.2.2.2 Background

Nb₃(Al, Ge) has the highest T_c for a material made using conventional bulk methods, arc-melting in this case. It was first formed by the Bell Lab group,⁽²⁾ and later, by many other groups using a variety of methods, including sputtering⁽³⁾ and electron beam co-deposition.⁽⁴⁾ The latter work, which will be referred to as the GA (General Atomic) work, is of most relevance since the same technique will be used here. This work showed that essentially bulk T_c was found in material that was nearly, if not entirely single phase.

The conditions and results obtained in the GA work were as follows:

Rate of Deposition: ~ 250 Å/sec.

System Pressure: ~ 10⁻⁶ Torr.

Substrate: Nb ribbon, 2 mm wide and either 44 or 76 μm thick. Substrate ribbon passed around a number of pulleys and idlers. Ribbon was

heated by passing current through the pulley wheels. Temperature was determined by calibration of the current vs the indicated reading of a thermocouple attached to the ribbon.

Substrate Temperature: The as-deposited T_c was found to peak at a substrate temperature of 1150°C. Because of this peak, a variation in the substrate temperature will result in a scattering of T_c to lower values (see Figure 2, Ref. 4).

Thickness of Deposit: The as-deposited T_c decreased for thickness below 0.3 μm . Above this value, up to 2.3 μm , the T_c was constant.

T_c Measurement: The values discussed are the midpoint of a resistive measurement, which on the much lower resistance Nb substrate indicates only the extreme lower end of the transition. The reduction in T_c is estimated to be $\sim 1.0\text{K}$.

As-Deposited T_c and Phase Identification: Figure 2 shows a segment of the ternary isothermal phase diagram for 1200°C with the hypothetical assignments of phases. The boundaries were established using X-ray diffraction and the result of Muller's study.⁽⁵⁾ Because of μ -probe uncertainties and the difficulty of the establishment of phase boundaries using the disappearing phase method (X-ray diffraction), the phase boundaries shown are probably uncertain to several atomic percent. On the basis of recent work at Stanford and a recent phase diagram of Nb_3Al ,⁽⁶⁾ the phase boundary at the Nb-Al side is near 22 at. % Al. Most likely, the boundary from there is probably $\sim 2\%$ higher in Nb across the ternary. The as-deposited T_c 's are also indicated on Figure 2 at their corresponding compositions. Only about 50% of the data points are shown in the area of highest T_c , and none outside that area (see Figure 1, Ref. 4 for a larger sampling. Note that the compositions are in wt. % there). There are several points to note: In the single phase Al5 region one conventionally expects to see that the T_c would increase towards the Al5 phase boundary as the Nb concentration decreases towards 75 at. %. If the phase extends below 75%, T_c should decrease (e.g., V_3Ga). In Figure 2 there is no evidence of this, although two of the highest T_c 's

Fig 1

are close to the phase boundary (as is the highest T_c after annealing — as discussed later). At least part of this lack of a trend can be assigned to the variation in T_c due to its sharp dependence on the substrate temperature, and the fact that a range of temperatures were used before this was fully realized.

A second point concerns the T_c values in the two phase (Al5 + Nb₅(GeAl)₃) and in the three phase (Al5 + Nb₅(GeAl)₃ + σ - Nb₂Al) regions. Conventionally one expects that the T_c in a multi-phase region to be constant along lines of equal composition tie-lines, until the concentration and grain size decrease to such a point that proximity effects become important. The value should be equal to the value at the Al5 phase boundary where the tie-line connects. This appears to be fairly well satisfied in Figure 2, considering the point discussed above.

Annealing Results: The arc-melted alloys in the literature had T_c onsets in the 18 to 18.5 range [Blaugher et al. (7) reported a midpoint of 18.0K for levitation melted material]. Long-term annealing increased the T_c onsets to 19.5 to 20.7, and midpoints of 19.8 and 20.1 were reported. The temperature-time used ranged from 750°C — 500 hours to 950°C — 72 hours. (8)

The GA thin films were judged to be already homogeneous, so a lower temperature of 700°C was used. The time for a maximum T_c was found for a limited number of specimens, indicated by the points A to E in Figure 2. Generally T_c was still increasing after 200 hours, although the specimen with the highest T_c peaked at 40 hours (pt. A). Generally the T_c increased by ~ 1.0K, with the largest increase of 1.25K. The highest T_c midpoint was 18.7, with 90% of the resistance restored at 19.0K. As discussed earlier, the Nb substrate shunting effect is such that the real T_c is estimated to be ~ 1K higher.

1.2.2.3 Present Sample Fabrication

Deposition Conditions:

Rate of Deposition: 30 Å/sec

System Pressure: 1×10^{-7} Torr

Figure 3 - Ternary Nb-Al-Ge System: Compositions in Stanford Runs
 G 80-33: $T_s = 1030^\circ\text{C}$, anneal $800^\circ\text{C}/20$ mins. G 80-45:
 $T_s = 1050^\circ\text{C}$.

10

Substrate:	Single crystal polished sapphire
Substrate Temperature:	1030 to 1050°C
Thickness of Deposit:	1.0 μm
T_c Measurement:	Mostly inductive, T_c^L ; certain selected specimens measured using four-point resistance determination, T_c^R .

Compositions Deposited: The earlier GA work used three sources arranged roughly as the vertices of a regular isosceles triangle. The small substrate area resulted in a specimen with essentially a single composition. In the present work the sources are arranged in a straight line (see Ref. 8 for dimensions), with the order being Nb, Al, Ge (the Nb is in the "Nb source" of Ref. 8).

The substrates are $\frac{1}{4}$ " x $\frac{1}{4}$ " square sapphire. These are arranged in three rows of ten each, with the direction of each row parallel to the line connecting the three sources (in Ref. 8, the phase spread orientation). Reference 8 discusses the spread in composition for binary and ternary depositions. Figure 3 shows the compositions of the specimens to be discussed in this report. The analysis made above of the GA work was used in selecting the region to study. (The scales and ranges of composition are the same for Figures 2 and 3.) The specimens along each row are numbered 1 through 10 as shown. The μ -probe was used to determine the compositions along each of the rows, which were deposited on sequentially using a movable mask. A change in the rate settings permits a change in the compositions spanned on each of the three rows.

Two separate runs have been done, G-80-33 and G-80-45. Run 33 produced two usable rows, A and C, and 45 produced rows, A, B, and C. The indicated substrate temperature of 33 was 1030°C, and of 45 was 1050°C.

1.2.2.4 As-Deposited T_c

Figures 4 to 8 show the T_c for each of the samples measured along each of the rows indicated in Figure 2. The specimens have been labeled as slide number for ease of illustration. The corresponding composition can be found from Figure 3. The majority of the data are T_c^L

G-80-33

Row A

SQUARE 10 X 10 TO THE INCH AS 3807 GO

T_c ,
K

18

17

16

15

14

13

12

11

Figure 4 - T_c vs sample position.

12

1 2 3 4 5 6 7 8 9 10

SLIDE No

T_c^R

T_c

x

U

A

G-80-45

Row A

SQUARE 10 X 10 TO THE INCH AS 0001-50

T_c^L
K

T_c^R

T_c^R

T_c^R

Repeat

Repeat

Figure 5 - T_c vs sample position.

13

SLINE No

G-80-33
Row C

T_c
K

16

15

14

13

12

11

10

9

Figure 6 - T_c vs sample position.

14

1^c 2^c 3^c 4^c 5^c 6^c 7^c 8^c 9^c 10^c

SLIDE NO. # Sam

SQUARE 10 X 10 TO THE INCH AS 0807 CU

G-80-45

Row C

SQUARE 10 X 10 TO THE INCH AS OBT. CO

T_c^L
(K)

Figure 7 - T_c vs sample position.

SLIDE No.

B *sign* G-80-45

SUAPR 10 X 10 TO THE IN H AS 9807 50

T_c
L
K

Figure 8 - T_c vs sample position.

values; the few T_C^R are indicated. The bars show the 5 to 95% values. A double line indicates an apparent break in the transition, and if one of the parts of the transition is significantly larger, a "L" is shown.

To compare with the previous GA data, in which the midpoint of the resistive (shunted) transition is shown in Figure 2, one uses the midpoint of the T_C^R , if available, or the upper onset of the T_C^L .

A number of points in regard to the T_C data follow:

1. In the high Nb single-phase Al5 region the T_C shows the expected linear increase as the Nb poor phase boundary is approached.
2. Three out of the five rows show a T_C max for the specimen believed to be at the phase boundary (indicated by the arrow at the bottom of each figure). The other two show a slight decrease in T_C , followed by a continued increase in T_C . A T_C increasing into the two-phase region has been noted in the work on Nb_3Al .⁽⁹⁾
3. In Figure 9 the maximum T_C at the assumed phase boundary, and the maximum T_C in the three-phase region, have been indicated on top of the data shown previously in Figure 1. One notes that in the three-phase region the present T_C 's are ~ 1K lower, and in the assumed single-phase Al5 region they are ~ 1.8K lower. Some of the difference may be due to the lower T_s used in the recent work (about 100°C, but calibration could be very different).

1.2.2.5 Discussion and Conclusion

Annealing experiments are in progress. The results of these, and of X-ray structure determination, will help to clarify the situation. It appears at this point that a higher substrate temperature is desirable in order to achieve results comparable with the previous GA results.

Figure 9 - Ternary Nb-Al-Ge System: New data showing the A15 Phase Boundary.

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1.2.2.6 Future Work

In the next quarter the annealing experiments on these specimens will be analyzed, and some X-ray structure determinations made. A new substrate holder is nearing completion which will allow a higher substrate temperature, and a better determination of the actual temperature than up to now. With this, runs will be made at ~ 1150 to 1200°C , and if indicated, higher. Measurements of T_c^* will be made on selected specimens to test if this system is homogenous.

1.2.3 Beam-Annealing of Nb₃Ge (Westinghouse)

1.2.3.1 Objective

The objective of this task is to establish whether the pulsed laser and/or electron beam annealing of Nb₃Ge tape conductors can lead to a significant enhancement of $J_c(T)$ without any marked deterioration of T_c^* . Preliminary results obtained in 1980 suggested that such an enhancement can occur upon laser annealing (LA) but only in a small and undetermined volume fraction of the ribbon superconductor.⁽¹⁰⁾ The enhancement was attributed to the nucleation of additional second phase Nb₅Ge₃(σ) flux pinning centers. In the light of the above results it thus became necessary to: (a) Develop electron microscopic techniques that would permit distinguishing between various microstructural layers in the film, (b) Determine the volume (thickness) of the improved layer fraction where nucleation occurred, (c) Establish whether significant volume fractions can be modified in films of practical thickness, in the range of 2 to 5 μm (use electron beam annealing for this purpose).

1.2.3.2 Microstructure of Laser Annealed Films

Transmission electron microscopy (TEM) was used to investigate the near-surface microstructure of a 5 μm thick film (459-0) that was laser annealed at a pulse energy of $E_p = 2\text{J}/\text{cm}^2$. While the reference film that was not annealed showed the typical crystalline microstructure and the Al₅ structure diffraction pattern, [Figure 10(a)] the surface of the annealed sample was clearly amorphous [Figure 10(b)] due to quick resolidification after melting. These results directly confirm the earlier

a. Not subject to laser annealing (reference sample).

b. After laser annealing, $E_p = 2\text{J/cm}^2$

Figure 10 — TEM characterization of the Nb-Ge surface layer;
sample 459-0.

data obtained by X-ray diffraction and measurement of J_c in very thin Nb-Ge films.⁽¹⁰⁾ Observation of a boundary region between the amorphous and crystalline layer also indicated the presence of very small grains and/or precipitates that could be the cause of J_c -enhancement. This observation is, however, not yet definitive since such grains or precipitates might represent an artifact of the TEM sample preparation by ion milling. Further study of this question is in progress.

1.2.3.3 Effect of Annealing vs the Film Thickness

Present lack of high-temperature heat conductivity and reflectivity data for Nb₃Ge makes impractical the calculation of energy deposition and temperature depth profiles. An experimental approach to roughly estimating the thickness (volume) of the layer in which beneficial Nb₅Ge₃ nucleation should have occurred consisted of annealing a series of films having various thicknesses, h , in the range from 10^3 to 5×10^4 Å. While the measurement of J_c gives an average over the whole volume of the sample there should be an intermediate film thickness range where the observed J_c -enhancement is the highest. Indeed, as shown before,⁽¹⁰⁾ at the pulse energy of $E_p = 2\text{J/cm}^2$ the thinnest films (one to several thousands angstroms thick) are melted and converted into amorphous or disordered phases having $J_c = 0$. On the other extreme, in the very thick films the J_c -improvement must be very small due to averaging over the entire volume. Unfortunately, the only series of Nb-Ge samples available for this investigation was deposited on Hastelloy B substrates, in contrast to the thin samples used earlier that were deposited on Ta and Nb ribbons in order to avoid substrate elements diffusing into the film. It was hoped, however, that the diffusion effects will be small. Also, limited amount of material available did not permit using reference samples nearly identical to those annealed. Instead, the J_c in each sample was determined twice: before and after annealing. For each J_c measurement samples were coated with a sputtered copper layer. For LA this copper had to be stripped by etching and a new layer deposited afterwards.

Two laser energy levels were used:

1. 1 pulse $E_p = 2\text{J/cm}^2$
2. 3 pulses $E_p = 1\text{J/cm}^2$.

Rutherford backscattering profiles of Bi^{209} implanted marker redistribution showed that (1) produces melting at least $10^3 \text{ \AA}$ deep while (2) produces no melting. (11)

The results of J_c measurements are shown in Figure 11 where the J_c change, $(\Delta J_c/J_c)$, is plotted vs the film thickness. In contrast to earlier data (1) the J_c -enhancement is not observed. Over the whole thickness range the $(-\Delta J/J_c)$ decreases nearly linearly with increasing film thickness. The $\Delta J_c/J_c$ values of Figure 11 are almost field independent and represent an average over the field range of 2 to 15 tesla at $T = 4.2\text{K}$. Figure 3 shows also that lower pulse energy produces lesser J_c -deterioration. These unexpected results prompted an immediate check of (a) the effect of copper stripping and recoating and (b) the Hastelloy substrate diffusion effects.

Table 1 shows that copper stripping and resputtering using the same unannealed samples produced a random and, occasionally, significant deterioration of the critical current I_c .

Table 1
Effect of Copper Coating on Critical
Current in Nb-Ge Tape Samples

Sample	I_c (A) Measured		
	After first coating	After etching Cu and recoating	% Change
435 B1	235	215	-9
435 D1	242	190	-21
435 F3	202	120	-41*

* > 50% at lower fields.

Figure 11 — Change in critical current density vs the film thickness for two pulse energy levels.

The cause of the I_c degradation is not known at present. The effect may be simply due to repeated handling. In any case, however, the present I_c characterization method that requires copper-coating must be changed to get unambiguous data.

The diffusion of Hastelloy B substrate elements (Ni, Mo, Fe) into Nb-Ge films was investigated using the EDX capability of a scanning electron microscope and by the Auger spectroscopy. Earlier Auger work identified nickel as the substrate element most readily diffusing into Nb-Ge.*

The EDX energy spectra up to 10 keV permit an easy identification of Ni and Fe. Samples several microns thick that were not subject to LA showed no or very slight presence of Ni and Fe in the probed near-surface layer (1 to 2 μm thick). Figures 12(a) and 13 show that after a single laser pulse, $E_p = 1$ and 2 joule/cm² the Ni and Fe peaks are still weak compared to the Ge peak, but after five 1 joule/cm² pulses they become quite strong [Figure 12(b)]. This suggests that LA can result in a significant diffusion of Ni and Fe into the bulk of the film. The above result was so far verified by probing five different areas of the tape specimen No. 435E but it requires further verification after additional samples subject to multiple pulses will become available.

Auger spectroscopy was used to determine depth profiles of nickel diffusing into thinner Nb-Ge films (a) as grown and (b) after LA using a single pulse $E_p = 2$ joule/cm².

Table 2 shows the results. A single pulse LA produced significant diffusion of Ni into a 2500 Å thick Nb-Ge film where the liquid/solid interface that existed during annealing was close to the film/substrate interface. However, in a 16,000 Å thick film, where these interfaces were more than a micron apart the LA did not result in any significant change in the Ni profile. It is difficult, therefore, to ascribe the 70% deterioration of J_c in this film (Figure 11) to the effect of diffusion. The lack of J_c enhancement in the sample series of Figure 11 cannot be fully

* J. Schreurs, unpublished results (1976).

Figure 12 — EDX spectra for $\sim 5 \mu\text{m}$ thick samples laser annealed at $E_p = 1 \text{ joule/cm}^2$. (a) Single beam pulse, (b) five beam pulses.

Figure 13 — EDX spectra for an $\sim 5 \mu\text{m}$ thick sample after annealing with a single pulse, $E_p = 2 \text{ joule/cm}^2$.

explained by either substrate diffusion or copper-coating effect, and the inconsistency between present and earlier data⁽¹⁰⁾ requires further clarification.

Table 2
Depth Profiles of Ni in Nb-Ge Films

Pulse Energy E_p , J/cm ²	~ 2500 Å thick film 459-U		~ 1.6×10^4 Å thick film 459-R	
	Approximate Depth in Film Å	At. % Ni	Approximate Depth in Film Å	At. % Ni*
0 (as-grown)	200	0	100	0
0 (as-grown)	10,300 (substrate)	mostly Ni (spec.: 60 wt %)	≤ 10,300 11,300	0 ~ 4
2	200	8	200	0
	600	9	6,000	0
	1,100	28	8,000	0
			11,000	~ 4

* Normalized to 100 at. % Nb + Ge + Ni.

1.2.3.4 Microscopic Observation of Surface Melting

Earlier data indicated that LA results in Nb-Ge surface melting at $E_p > 1.5$ J/cm².⁽²⁾ However, present scanning electron microscopy (SEM), and scanning transmission electron microscopy (STEM) work shows nonambiguously that melting can occur even at $E_p = 1$ J/cm². Figure 14 compares the SEM surface photographs before LA, and after single 1 J/cm² and 2 J/cm² pulses. Clearly, already $E_p = 1$ J/cm² produces surface glazing while $E_p = 2$ J/cm² results in a completely melted surface. Figure 15 shows a STEM photograph and the electron diffraction pattern of the amorphous surface layer produced by applying $E_p = 1$ J/cm². The layer was EDX analyzed at points A and B. Figure 16 shows the corresponding EDX spectra indicating that the thin amorphous layer is predominantly Nb-Ge and not a redeposited

0J/cm²

1J/cm²

2J/cm²

Figure 14 — SEM surface photographs at various laser pulse energy levels;
X3200.

x50,000

Figure 15 — STEM photograph of amorphous surface layer produced by $E_p = 1\text{J}/\text{cm}^2$; at points A and B EXD analysis was performed (see Figure 16).

NBGE435C2/A

PR= S 70SEC 0 INT

V=512 H=20KEV 1:30 AQ=20KEV 10

NB,GE

NBGE435C2/B

PR= S 75SEC 0 INT

V=512 H=20KEV 1:30 AQ=20KEV 10

NB,GE

Figure 16 — EDX spectra from points A and B in Figure 15.

substrate material (Ni, Fe) that accumulates as a result of ion-milling of the sample. In light of the above results, there is no doubt that even at $E_p = 1 \text{ J/cm}^2$ deleterious resolidification strains will be induced in the film. Additional LA experiments at $E_p < 1 \text{ J/cm}^2$ are necessary to determine the true Nb-Ge melting threshold. The SEM represents a simple and effective tool for such an investigation. One can infer from Figure 11 that the depth of melting at $E_p = 1 \text{ J/cm}^2$ is less than at $E_p = 2 \text{ J/cm}^2$. The threshold may be just slightly below 1 J/cm^2 .

1.2.3.5 Directions of Future Laser Annealing Work

Results presented above show the need for methodological improvements in the beam annealing work. The problem of the process optimization is more complicated than initially believed. The immediate objectives to be addressed in the third and fourth quarters of 1981 will be to:

1. Fabricate Nb-Ge samples on Ta and/or Nb substrates and limit the investigation to such samples (film thickness range of 10^3 to $10^5 \text{ \AA}$).
2. Characterize J_c by magnetization measurement of small, uncoated discs.
3. Repeat earlier LA experiments using the approach of (1, 2).
4. Determine the LA surface melting threshold using SEM and STEM.

1.2.3.6 Electron Beam Annealing Apparatus

A simple pulsed electron beam system described in the schematic diagram of Figure 17 was assembled to anneal Nb-Ge films. The general view of the system is shown in Figure 18. The films were to be up to $h = 5 \text{ }\mu\text{m}$ thick and therefore 10 keV electrons which would have a range of about $2.5 \text{ }\mu\text{m}$ were desired. The synthetic transmission line consisting of the three inductors and capacitors shown in Figure 17 will produce a voltage pulse of peak value $V/2$ where V is the voltage to which C is charged and width $\sqrt{LC}/2\pi$ seconds when the switch s is closed. The voltage pulse

Figure 17 — Schematic diagram of pulsed E-Beam system.

Figure 18 — General view of the E beam pulse annealing apparatus.

a. Voltage

b. Current

Figure 19 — Voltage and current pulses obtained upon closing the switch.

is shown in Figure 19(a). When the filament is cold, no current is measured by the shunt. When between 11 and 15 amperes flows in the filament sufficient electrons are emitted to produce the current pulse seen in Figure 19(b). Note that the ringing current (peaks 2 to 4) is accompanied by almost no voltage and therefore represents almost no power to the sample. The product of the current, voltage and pulse width represent in this example an energy density of 1 joule/cm² to the sample.

The initially assembled vacuum system with a mechanical roughing pump only provided a vacuum of 10⁻¹ to 10⁻² Torr. Preliminary experiments indicated, however that this was insufficient to prevent sample oxidation. The vacuum system was rebuilt, diffusion pump included and a satisfactory vacuum of 10⁻⁴ Torr obtained.

Preliminary experiments are being continued to determine the effect of the tungsten filament thermal radiation on the sample prior to the E-beam pulse application.

1.2.3.7 Directions of Future Electron Beam Annealing Work

In the next quarter the preliminary testing of the E-beam apparatus will be completed with additional modifications introduced as needed. Subsequently, the Nb-Ge melting threshold will be determined for various pulse energies and pulse widths. Measurements of J_c will be performed using the magnetization and transport techniques.

1.2.4 Modification of CVD Process (Westinghouse)

1.2.4.1 Objective

The objective of this task is to reactivate the CVD apparatus for Nb-Ge tape fabrication, to investigate process modifications leading to an improved Al5 phase homogeneity, higher T_c^* and $J_c(T)$, and to fabricate tape specimens for other tasks of the program.

1.2.4.2 Results

After the preliminary run reported in the First Quarterly Report minor modifications were introduced in the glassware of the reactor, essentially to restore the process conditions attained in 1977-1978, i.e., before the system became inactive. Subsequently, a systematic fabrication

experiment was performed at a deposition temperature $T_d \approx 900^\circ\text{C}$. A series of Nb-Ge tape samples were fabricated and characterized. The T_c 's in excess of 21K and low-field J_c 's exceeding 10^6 A/cm² at 4.2K demonstrated that the previous fabrication capability was restored. The system is now ready for the planned investigations.

1.2.4.3 Future Work

The first systematic modification of the apparatus to be introduced in the third quarter will consist of a different tape masking system that should reduce the destructive hydriding of Nb and Ta tape substrates without the necessity of substrate protection by copper-coating. After attaining this goal, work will concentrate on fabricating samples for beam annealing and mechanical properties improvement tasks.

2. PROJECT SCHEDULE AND FORECAST

The project is presently behind schedule. The fabrication of the vessels for dielectric tests on LH_2 has been delayed because of problems in passing the X-ray inspection. The fabricator has subcontracted this task to another fabricator with the necessary equipment. It is now expected that the fabrication of large vessels and their auxiliary equipment will be completed in July-August 1981. All other work related to the dielectric tests of LH_2 (Part I of the program) was suspended due to the lack of additional funds for this part of the program and the uncertainty concerning the program continuation in FY 1982.

The Part II of the program (concerned with superconducting materials) progressed satisfactorily with only minor delays resulting from technical difficulties encountered in the course of the investigation. The apparently most significant delay occurred in Task 1.2.5: "Improvement of Mechanical Properties of Nb_3Ge Tapes." This task depends upon future results of Task 1.2.4 and a rapid progress is anticipated for the fourth quarter of 1981 (and beyond) if the program continues in FY 1982.

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