DESIGN OF A TUNABLE HIGH-Q SUPERCONDUCTING NOTCH FILTER

by

Charles M. Falco, R. T. Kampwirth,
C. S. Pang, and Ivan Schuller

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Solid State Science Division

October 1978

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ABSTRACT

We report on the design of a tunable high-Q superconducting notch filter. The filter is designed to be manufactured from high T_c superconductors (Nb_3Sn , Nb_3Ge) made by high-rate magnetron sputtering on sapphire substrates.

We report on considerations relating to the geometry of the various elements, on holder materials for the cryostat, on studies relating to the preparation of suitable high T_c materials, and on the photoetching procedures for the filter elements.

A. Filter Design

I. INTRODUCTION

In general the filter design aspect can be broken down into 3 major categories: cryogenics, actual filter design and electronics. Roughly speaking the cryogenics has to include a variable temperature cryostat of large enough size to house the filter, appropriate thermometers, vacuum pumps and vibration isolators to decouple mechanical vibrations from the cryostat. The actual filter design includes selection of support materials for the substrate that have the appropriate mechanical, cryogenic and electrical properties and the design of low temperature vacuum sealed feed-throghs for frequency tuning. The electronics deals with studies relating to coaxial lines for signal transmission, electronic apparatus and shielding from environmental rf noise. Most of the design and description of materials is appropriate for both the flat and the cylindrical geometries. To be more specific, we will use as a prototype one of the flat geometry designs made from niobium films deposited on fused quartz substrates.

II. CRYOGENICS

Fig. 1 shows a sketch of the glass dewar that will accomodate the variable temperature cryostat. The large (4") I.D. of the helium dewar is necessary to accomodate the 2" diameter substrates and their holders. Figs. 2 and 3 show the table and plate from which the dewar is suspended. In order to avoid coupling of vibration from the pumps to the cryostat the 4" pumping lines will be fed to the dewar through the vibration decoupler shown in Figs. 4, 5, 6, and 7. The effect of the vibrations on the performance of the filter will be discussed in detail in Section A-IV-4.

The cryostat consists of a double walled variable temperature insert for the helium dewar. The temperatures are measured using Ge thermometers and controlled to better than 1 mK by either requesting the bath pressure or by using a SHE model ATC temperature controller.

III. ACTUAL FILTER DESIGN

An equivalent circuit for the planar filter is shown in Fig. 8. The various elements are evaporated on 2" diameter sapphire substrates and are shown in Figs. 9, 10, 11 and 12. The calculations performed in Section A-IV-2 show that the spacing between plate C_2 and L_2 should be in the cm range and the spacing between L_1 and L_2 should be variable between .001" and $\sim$.060" in order to be able to span an appreciable fraction of the frequency range as well as the required notch shape over which the final filter operates. In brief, the substrates should be mounted on holders made out of a material of low dielectric constant and low loss tangent in order to avoid appreciable distortions of the fringing fields as well as the dielectric losses which may limit the Q. The holder of material should have thermal expansion coefficients closely matching that of sapphire; furthermore, it should be machinable and should be mechanically rugged.

Fig. 1 Helium dewar

Fig. 2 Support table

Fig. 3 Cryostat platform

Fig. 4 Cryostat-probe coupling and vibration decoupler.

Fig. 5 Vibration decoupler detail.

Fig. 6 Positioning of support rods for the decoupler.

Fig. 7 Cryostat-probe coupling detail.

Fig. 8 Basic circuit of the notch filter.

$L_1 = 40 \mu\text{H}$
 $L_2 = 211 \mu\text{H}$
 $M \cong 24 \mu\text{H}$ FOR 1cm SPACING
 $C_2^{(o)}$ = OUTER CAPACITOR
 $C_2^{(i)}$ = INNER CAPACITOR
 R_C = POSSIBLE LOSSES IN / BETWEEN
 $C_2^{(o)}$ AND/OR $C_2^{(i)}$
 R_2^L = POSSIBLE LOSSES IN L_2

Fig. 9 The actual circuit design

SPIRAL INDUCTOR L_1

0.002" line on 0.0071" centers
Approx. 25 turns
Approx. $40\mu\text{H}$

Fig. 10 Primary induction coil of the actual circuit.

SPIRAL INDUCTOR L_2

0.002" line on 0.003" centers
Approx. 58 turns
Approx. $211\mu\text{H}$

Fig. 11 Secondary induction coil with ends terminated on the capacitor plates.

CAPACITOR C_2 (Bottom Plate)

Fig. 12 Capacitor plates.

Roughly speaking the Q of the circuit is given as

$$\frac{1}{Q} = \frac{1}{Q_R} + \frac{1}{Q_D} + \frac{1}{Q_r} + \frac{1}{Q_c}$$

where Q_R relates to the resistance in the tank circuit, Q_D relates to the dielectric losses which are proportional to the loss tangent of the dielectric material in the circuit, Q_r relates to the radiation loss which can be minimized if good shielding is provided, and Q_c is related to the coupling loss.

Table I shows a list of the thermal expansion coefficients, dielectric constants and loss tangents of relevant holder materials.

TABLE I

Construction Materials For Filter Support

Material	Room Temperature Thermal Expansion Coefficient (/ °C)	Dielectric Constant (CGS units)	Loss Tangent	Machinability	Mechanical Properties
<u>Nb</u>	7.1×10^{-6}				
<u>Sapphire</u>		9.0	.0002		
Parallel to C axis	6.66×10^{-6}				
60° to C axis	5.8×10^{-6}				
Perpendicular to C axis	5.0×10^{-6}				
<u>Boron Nitride</u>	8.0×10^{-6}	5.0	.0002	good	soft
<u>Macor</u> (Corning glass ceramic)	9.4×10^{-7}	6.0	.0002	good	very good
<u>Epoxy</u>					
Stycast 2850GT	29×10^{-6}	5.9	N.A.		good thermal and Mechanical shock properties
Catalyst 24LV					

According to Table I there are two choices for holder materials: Boron Nitride and Macor. Both have thermal expansion coefficients close to that of sapphire, suitable mechanical properties, high dielectric constants and low loss tangents.

In an attempt to simplify the design problem, Nb was deposited on high temperature ($\sim 500^\circ\text{C}$) Boron Nitride substrates. The resulting films were of very poor quality so that no further work was conducted using Boron Nitride as a substrate material.

The filter assembly concept is shown in Figs. 13, 14. Capacitor plate C_2 is adjusted by means of a Mitutoyo-297-101 nonrotating micrometer head. The movable shaft is sealed to the feedthru with a double O-ring. Inductors L_1 and L_2 can be kept separate by using suitable spacers and a dielectric can be inserted between L_2 and C_2 in order to increase the frequency range.

IV. ELECTRICAL CONSIDERATIONS

1. Calculation of Mutual Inductance Between L_1 and L_2

To make an approximate calculation of the mutual inductance between L_1 and L_2 we will calculate the mutual inductance between the innermost set and outermost set of coils. These values will then be averaged and multiplied by the total number of turns. For two coils of radius a and separation d , the mutual inductance is given by $M_0 = fa$ where f is a function of $\frac{d}{2a}$ and is tabulated numerically by Grover table 16,*

$$M_{\text{total}} = M_0 \times N_1 \times N_2 \text{ and } K = M/\sqrt{L_1 L_2}.$$

Table II shows the various parameters and the average mutual inductance for two different separations.

TABLE II

d [cm]		a [cm]	d/2a	f [$\mu\text{H}/\text{cm}$]	M_0 [μH]	$\bar{M}_0$ average [μH]	M total [μH]
0.5	innermost	1.4936	.168	.01580	.02368	.02961	42.93
	outermost	1.9291	.130	.01842	.03553		
1.0	innermost	1.4936	.3336	.008213	.011231	.01685	24.43
	outermost	1.9291	.2592	.011086	.02139		

* F. W. Grover, Inductance Calculations, p. 83, Dover Publications. New York, 1962.

As shown in Table II the coupling constant can be easily adjusted to be in the range $0.25 < K < 0.5$ using reasonable separations between L_1 and L_2 .

2. Calculation of Capacitance C_2

The calculation of the capacitance is based on the fact that C_2 is made of two circular capacitors in parallel. For a parallel plate capacitor

$$C = \frac{\epsilon_0 A}{d} \text{ where } \epsilon_0 \approx 8.854 \times 10^{-12} \text{ C}^2 \text{J}^{-1} \text{m}^{-1}$$

for vacuum. Table III shows the relevant parameters for the two capacitors, for two different separations.

TABLE III

$d = 1\text{cm} = 10^{-2}\text{m}$					$d = 0.1\text{cm} = 10^{-3}\text{m}$	
r_1 [cm]	r_2 [cm]	A [10^{-4}m^2]	C [pF]	C_{total} [pF]	C [pF]	C_{total} [pF]
inner 0	1.4986	7.055	0.625	0.315	6.25	3.15
outer 1.929	2.451	7.1828	0.636		6.36	

These capacitances can be increased by at least a factor of 100 using the appropriate dielectric filling. However, a careful selection of this filling material should be made such that the loss tangent does not lower the Q significantly.

3. Resistance

For an acceptable notch shape $L_2 = 211 \mu\text{H}$, R_2 must be less than 0.32Ω (See Fig. 15).

Assuming resistive transitions of maximum 0.5 K (which can be easily achieved in our sputtering systems) the resistance R_2 and R_c (See Fig. 8) should be smaller than a few $\text{n}\Omega$ at temperatures below 18°K for Nb_3Sn below 21°K for Nb_3Ge and below 7.5°K for Nb . Thus the resistance is not a limiting factor on the notch shape and Q .

Fig. 13 High temperature probe and filter housing.

Fig. 14 Cryostat top plate with micrometer head for tuning control.

Fig. 15 Basic circuit design limits for desired notch shape.

Fig. 16 Capacitor plates with dielectric showing lateral vibration.

4. Calculation of Vibrations Effects on Capacitance

For parallel plates, $C = \frac{\epsilon A}{d}$, and $\epsilon_o \approx 8.854 \times 10^{-12} \text{ C}^2 \text{ J}^{-1} \text{ m}^{-1}$. Assume a filling plug is used with $\epsilon = 100$ and filling factor .5 as shown in Fig. 16, $C = 3.12 \times 10^{-13} \approx 0.3 \text{ pF}$ and the resonant frequency f_o will be 19.6 MHz. The total volume of the dielectric is 3.53 cm^3 ($3.53 \times 10^{-6} \text{ m}^3$). When the dielectric vibrates in one plane, the filling factor changes. As shown in Fig. 16(b), the effective area of the dielectric within the capacitor is

$$A_{\text{diel}} = L(L - \delta)$$

The effective area of the "vacuum" is $L\delta$ so that

$$C_{\text{diel}} = \frac{\epsilon A_{\text{diel}}}{d} \times \frac{t}{d} = \frac{\epsilon L(L-\delta)t}{d^2} \quad (= 19.5 \text{ pF for } \delta = 1\text{cm})$$

and

$$\begin{aligned} C_{\text{vac}} &= \frac{\epsilon_o A_{\text{vac}}}{d} + \frac{\epsilon_o A_{\text{diel}}}{d} \frac{(d-t)}{d} \\ &= \frac{\epsilon_o L\delta}{d} + \frac{\epsilon_o L(L-\delta)(d-t)}{d^2} \quad (= 0.23 \text{ pF} + 0.20 \text{ pF for } \delta = 1\text{cm}) \end{aligned}$$

To calculate the vibrational effect, we need to differentiate to get

$$\begin{aligned} \frac{\partial C}{\partial \delta} &= -\frac{\epsilon L t}{d^2} + \frac{\epsilon_o L}{d} + \frac{\epsilon_o L(t-d)}{d^2} \\ &= \frac{\epsilon_o L}{d^2} [\epsilon t + d + (t-d)] = \\ &= \frac{\epsilon_o L}{d^2} t(\epsilon+1) = 1.189 \times 10^{-9} \text{ F/m} \approx 1.19 \text{ pF/mm} \end{aligned}$$

The capacitance changes by approximately 6% per mm of travel. The design criteria dictate that $R_2/L_2 \leq 1.6 \times 10^3$ over the intended tuning range so that

$$\begin{aligned} Q = \frac{\omega L}{R} &\geq 7.85 \times 10^3 \text{ @ } 2 \text{ MHz} \\ &\geq 1.17 \times 10^5 \text{ @ } 30 \text{ MHz} \end{aligned}$$

For $L_2 = 211 \mu\text{H}$ we have: $C_{2\text{MHz}} = 3.00 \times 10^{-11} = 30 \text{ pF}$

$$C_{30\text{MHz}} = 1.33 \times 10^{-13} = 0.13 \text{ pF}$$

At 2MHz:

$$\frac{1}{Q_2} C_2 = 3.82 \times 10^{-15} \text{ F} \times \frac{\partial \delta}{\partial C} = 3.21 \times 10^{-6} \text{ m} = 3.2 \mu\text{m}$$

At 30MHz:

$$\frac{1}{Q_{30}} C_{30} = 1.14 \times 10^{-18} \text{ F} \times \frac{\partial \delta}{\partial C} = 9.56 \times 10^{-10} \text{ m} \approx 9.6 \text{ \AA}$$

The above calculation shows that for the vibration of dielectric to alter the capacitance by less than $1/Q$ requires vibration amplitudes of less than $3 \mu\text{m}$ at 2MHz and $10 \text{ \AA}$ at 30 MHz. Actually the resonant frequency ($\sim C^{1/2}$) is the important quantity allowing, respectively, $10 \mu\text{m}$ and $100 \text{ \AA}$ vibration amplitudes. This indicates that, especially for the higher frequencies, a filter geometry with a large immunity to vibration must be used.

Similarly, to estimate the change of the filter performance due to vertical vibration of the capacitor plates, we consider the effective capacitance in the tank circuit shown in Fig. 8.

$$C = \frac{C_2^i C_2^o}{C_2^i + C_2^o} = \frac{\epsilon_o}{d} \left(\frac{A_i A_o}{A_i + A_o} \right)$$

where ϵ_o is the permittivity in vacuum, d the separation spacing between two capacitor plates, and A the area of the plate. The change of capacitance due to the vibration is

$$\frac{\partial C}{\partial d} = \frac{-\epsilon_o}{d^2} \left(\frac{A_i A_o}{A_i + A_o} \right)$$

The notch frequency of the filter is given as

$$f_o = \frac{1}{2\pi \sqrt{L_2 C} \sqrt{1-K^2}}$$

which can be approximated to $1/2 \pi \sqrt{L_2 C}$ for the K used in the present design. From these equations the effect of detuning can be calculated. The tolerable vibration is given as

$$d_T = \frac{C}{Q} / \frac{\partial C}{\partial d} .$$

The major element parameters and tolerable vibration magnitudes are listed in Table IV for two filters at different L_2 and four frequencies.

TABLE IV

L_2 (μH)	f_o (MHz)	C (pF)	Q	d^* (m)	$\frac{\partial C}{\partial d}$ (pF/mm)	d_T (mm)	R_2 (Ω)
211	2	30	7.9×10^3	0.10×10^{-3}	300.0	0.013×10^{-3}	0.34
	4.47	6	1.7×10^4	0.53×10^{-3}	11.3	0.031×10^{-3}	0.34
25	4	63.3	1.6×10^4	0.05×10^{-3}	1266.0	0.0002×10^{-3}	0.04
	30	1.13	1.2×10^5	2.79×10^{-3}	0.4	0.024×10^{-3}	0.04

* Calculated base on the effective area $A = \frac{A_i A_o}{A_i + A_o} = \frac{7.055 \times 7.183}{7.055 + 7.183} \times 10^{-4}$

$$= 3.56 \times 10^{-4} \text{ m}^2$$

The calculations showed that the mechanical stability of the mounting system is an important factor for long term stable tuning to be established. This effect is especially pronounced when the capacitor is tuned to higher capacitances.

B. SUPERCONDUCTING FILM PREPARATION AND

CHARACTERIZATION

I. SUBSTRATE

When choosing a substrate onto which films for the superconducting filter will be deposited, several factors must be taken into consideration. The films will be Nb and its compounds. A substrate should be chosen with a coefficient of thermal expansion α similar to that of the film, which for Nb and its compounds ranges from ~ 7 to 9×10^{-6} per $^{\circ}\text{C}$. This helps minimize thermal mismatch which can unduly stress the superconducting film causing deterioration of the superconducting properties and/or film lift off. Film lift off due to stress can be particularly troublesome during the pattern etching process. Filter performance is dependent on both the dielectric constant and the loss tangent and must be considered in choosing a substrate material.

Table V lists some of the substrate materials being considered along with the properties of interest. The maximum useable operating temperature is shown in this table since the A15 materials must be made at temperatures of about 800°C . The substrate must be able to withstand these temperatures without melting, outgassing, changing its electrical properties, etc. The dielectric constant and loss tangent are room temperature values. It is expected that the loss tangent should decrease with decreasing temperature although the only measured value at low temperatures is that of Lucalox at $\sim 15\text{K}$ with a loss tangent of 1.4×10^{-6} reported by Siegel, et. al(1). This is a two order of magnitude reduction over room temperature values. The dielectric constant should be maximized and the loss tangent minimized. This helps improve the Q of the circuit and the range over which it can be tuned.

II. SUBSTRATE HEATING

Since it is necessary to form Nb_3Sn in film form at substrate temperatures ranging from 700 to 900°C , a substrate heater capable of operating at these temperatures is required. For substrates of less than 1" diameter, a simple strip heater consisting of, e.g., 0.002" thick by 1" wide x 4" long tantalum foil capable of passing up to 100 Amps of ac current is perfectly adequate. However, when one or more discs of 2" diameter must be uniformly heated, the task becomes considerably more difficult due to radiation losses which go as T^4 and to the ability of heating materials to withstand these temperatures.

Two designs have evolved which are presently in the process of evaluation. The first involves radiation heating of a Cu or Ni plate using a 1200 Watt Quartz lamp. Preliminary tests indicate top surface temperatures in excess of 800°C are possible, however surface temperatures of the substrates themselves have been measured to be about 200°C lower at 1 KW input power. This heater works very well at about 500°C substrate surface

TABLE V

Substrate Materials

Material	Max Usable Temperature	β $\times 10^{-6}$	Dielectric Constant	25°C Loss Tangent
	°C	20 - 800°C per °C	relative to free space	$\times 10^{-4}$
<u>Fused Silica</u> (Suprasil)	800	0.55	4.0	0.38
<u>Al₂O₃</u> (Single Crystal)	2000	7 - 9	10.5 - 11.5	2.0
<u>Macor</u> Glass Ceramic	1000	12.3	5.9	40.0
<u>Lucalox</u> (Polycrystalline Al ₂ O ₃)	2040	7.0	10.0	1.5
<u>Rutile</u> (Polycrystalline TiO ₂)	1000	9.0	85	5.0

temperature and all the Nb on fused silica were made using this heater at 500°C.

The second design involves the use of thermocoax heating elements which have been brazed into grooves cut into the back side of a Ni or Cu plate as shown in Fig. 17. The top of such a plate has been machined flat and polished in order to lower its emissivity. The plate also contains a number of tapped holes for attaching substrates clamps. The entire unit can be placed in a radiation shield to reduce losses from the sides and bottom. This unit has been tested to about 750°C substrate surface temperature. The heater has failed at the joint between the external lead in wires and the Nickel Chromium heater wire. Methods of improving this joint are currently being tested. This type of plate heating appears to be the most promising and further refinements of the basic design are under consideration.

III. SUPERCONDUCTING FILMS AND THEIR PROPERTIES

Final specifications call for a superconducting filter operating in a closed cycle refrigerator at temperatures greater than 10K. To meet this requirement Nb₃Sn films with $T_c \sim 17.5K$ will be used. These films will be fabricated by magnetron sputtering in a vacuum system still being constructed and described elsewhere in this report.

a. Niobium. In order to evaluate the feasibility of making the various filter elements and measuring their properties, Niobium was chosen as the test material. It is a type II material like Nb₃Sn, has a T_c of $\sim 9.3K$, is easily made with our existing magnetron sputtering system, has a thermal expansion coefficient similar to Nb₃Sn, can be etched with the same solution as Nb₃Sn, can be made with a critical current density $> 10^6$ Amps/cm² at 4.2K and has a microstructure similar to Nb₃Sn. Table VI gives the deposition parameters of the material prepared for this program.

TABLE VI

Niobium Film Preparation Conditions

Target Material:	99.95% pure polycrystalline Nb
Sputtering Pressure:	3.5 mtorr
Sputtering Gas:	Argon
Target-Substrate Spacing:	3"
Substrate Temperature:	200 - 525°C
Deposition Rate	100 Å/sec
Target Power Input:	5.4 KW
Substrate Material:	Pyrex and Fused Quartz
Thickness:	500 nm

a.

b.

Fig. 17 Substrate heater using thermocoax heating elements embedded in Ni/Cu plate.

The vacuum chamber is pumped to $< 5 \times 10^{-7}$ Torr, followed by bringing the substrate up to the desired operating temperature. During this period the pressure can rise to as high as 1×10^{-5} Torr after which it decreases. Table VII gives a breakdown of the residual gases in the system just prior to introduction of the sputtering gas, showing clearly that the dominant gas is water vapor.

TABLE VII

Partial Pressures of Residual Gases in Vacuum System

Total Pressure = 2.1×10^{-6} Torr; $T_s = 495^\circ\text{C}$

Residual Gas Species AMU	Partial Pressure $\times 10^{-7}$ Torr
2	1.7
12	3.5
18	15.0
28	0.6
32	0.02
44	0.3
50-100	< 0.001

The presence of oxygen in the sputtering chamber due to background, dissociation of water vapor and contribution from the sputtering gas can have a significant effect on the quality of Nb films. Koch, et. al. (2) have shown the T_c of Nb is depressed by one degree per atomic percent oxygen in the film. A more sensitive measure of film quality is the residual resistivity just above T_c which can be expressed in terms of the resistivity ratio $\gamma \equiv \rho_{300}/\rho_{T_c}$. The larger this number, the lower the impurity content of the film and hence the better the film quality.

In Table VIII we have listed some of the electrical and superconducting properties of Nb films we have prepared by magnetron sputtering.

TABLE VIII

Properties of Sputtered Nb Films

ρ_{300}	10-13 $\mu\Omega$ Cm
ρ_{300}/ρ_{T_c}	6 to > 21
T_c	9.26 to 9.47K

To further elucidate the characteristics of these films we have looked at the microstructure by both scanning and transmission electron microscopy. The results are shown in Figures 18 and 19. As expected, Nb films tend to grow in a dense columnar structure with grain size going from $< 200 \text{ \AA}$ for films prepared at T_s of 200°C to $3000 \text{ \AA}$ for a T_s of 800°C .

b. Niobium Nitride. One of the many difficulties of preparing high T_c films has to do with substrate heating, particularly for non-planar geometries. NbN films having a bcc structure have been prepared by a number of authors at ambient temperatures (the equilibrium temperature reached by the substrate in the absence of direct substrate heating) using reactive sputtering. The process involves introducing a small partial pressure of nitrogen into the chamber during sputtering allowing a reaction between the niobium and nitrogen to form NbN on the substrate. These films have some rather interesting properties. T_c 's can range up to 15K , $H_{c2}(0) > 30$

Tesla residual resistivities $> 300 \mu\Omega\text{cm}$ and critical current densities in excess of 10^7 A/cm^2 at zero field and 4.2K . In addition, the bcc structure of these films which is more stable than the A15 structure of, e.g. Nb_3Sn , may prove more effective against degradation of the superconducting properties in intense radiation fields.

We have recently prepared NbN by d.c. magnetron reactive sputtering at a T_s of 200°C using an N_2 partial pressure of 1×10^{-3} Torr with an Ar pressure of 3.2×10^{-3} Torr. Power input was 0.86 KW . Using a weight gain method of measurement, film thickness was determined to be $1.76 \mu\text{m}$ and deposition rate 1.73 nm/sec (2.0 nm/sec/kW). The T_c of one of the films was measured using a 4 terminal resistive method and found to be 14.3K with a transition width of 0.9K . This temperature should be high enough to allow filters made with NbN to operate with a closed cycle refrigerator. Further evaluation of these films will be carried out.

c. Nb_3Sn . As discussed in the introduction of this section, Nb_3Sn is the superconductor agreed to for construction and evaluation of the final filter. The main advantage of this material is the high T_c obtainable, typically 17.5K to 18K in thin film form using preparation techniques such as magnetron sputtering. We have previously reported⁽³⁾ making films with T_c onsets of 18K , ΔT_c of 0.5K , $H_{c2}(0)$ of 23 tesla and self-field $J_c(4.2\text{K})$ of $1.5 \times 10^7 \text{ A/cm}^2$. These films show dense columnar growth structures with very smooth surfaces, an example of which is shown in the SEM micrograph of Fig. 20.

One of the most difficult aspects of making good Nb_3Sn films has to do with target construction. The very high target power inputs used in magnetron sputtering cause target heating producing preferential melting of Sn when it is tiled to the surface of a Nb base or mixed in the appropriate ratio with Nb powder in a composite powder target. The problem is particularly troublesome when using the down-sputtering geometry of our existing sputtering system. When the Sn begins melting it drips from the target onto the substrates, whether the target is tiled or mixed Nb-Sn

Fig. 18 Microstructure of sputtered Nb film

Fig. 19 Microstructure of sputtered
Nb film.

powder. Target composition changes with time resulting in decreasing film quality. To circumvent this problem we have devised a technique of powder target fabrication which ties the Sn powder up by reacting it with the Nb to form a higher melting point Nb-Sn compound. An example of a reacted Nb_3Sn powder target just prior to final machining is shown in Fig. 21. This procedure has proven very successful in producing Nb_3Sn films of very high quality. The main disadvantage is a tendency for the targets to crack during thermal cycling caused by the sputtering process. The target expands into the circumferential cooling ring during plasma heating causing target stress. Since Nb_3Sn is very brittle, the target can only relieve the stress by cracking. The cracking generally has no effect on the sputtering process itself unless pieces of the target begin to fall off as is the tendency in the down-sputtering configuration. Sputtering upward, as will be the situation in the new system, becomes a necessity for Nb_3Sn . We anticipate beginning this shortly after completion of the new sputtering system in the fall of 1978.

d. Nb_3Ge . To complete the discussion of materials suitable for filter construction, a few words about Nb_3Ge are in order. This compound is more difficult to form since the stoichiometric 3:1, A-15 phase which gives the 23K T_c is metastable at all substrate temperatures. However, under proper conditions the high T_c phase can be quenched in by a number of techniques including magnetron sputtering⁽⁴⁾. Using Nb_3Ge , it should be possible to operate the filter at temperatures as high as 20K. We are presently refining our film fabrication techniques to permit film formation over a 4" diameter area such that the film properties at all points within the area are uniform and possess T_c onsets $\geq 22\text{K}$ with ΔT_c 's $\sim 0.5\text{K}$.

Fig. 20 SEM micrograph of Nb₃Sn film indicates columnar growth structure.

Fig. 21 Reacted Nb-Sn powder sputtering target.

IV. ETCHED PATTERN DEVELOPMENT

The planar geometry decided on for the prototype filter is most conveniently made by etching the appropriate patterns in films of superconducting material. To test this concept we coated several 2 inch diameter discs of Pyrex and Fused Silica with $0.5\text{ }\mu\text{m}$ of Nb. These discs were spin coated at 5000 rpm for 25 sec with undiluted Shipley 1350J positive working photoresist. Resist thickness was estimated to be about $1.5\text{ }\mu\text{m}$. Following soft baking for 25 min. at 90°C , the films were exposed to light thru mask pattern D1. The exposure system consisted of a 500 W high pressure Hg-arc lamp with a lens system which afforded some degree of collimation. Initial efforts to expose the photoresist were made using the 4358 Å line in the Hg spectrum. However, the intensity was such that sufficient exposure could not be obtained in a reasonable period of time. When the entire ultra violet spectrum of the lamp was used, acceptable exposures were obtained in about 10 minutes. The resist was then spray developed for 90 sec with Shipley AZ-351 developer, rinsed with distilled water, dried and post-baked for 30 minutes at 130°C in a forced air oven.

Pattern etching was accomplished using a Nb etchant developed in our laboratory. It consists of 1 part HNO_3 , 1 part H_2SO_4 , 2 parts HF and 4 parts H_2O . Etching is done at room temperature. It takes approximately 10 sec to etch thru $0.5\text{ }\mu\text{m}$ of Nb. Patterns etched on Pyrex contained a number of defects. Typical examples are shown in Fig. 22. The black lines comprise the Nb film coil geometry and are typically about $35\text{ }\mu\text{m}$ wide. The light area in between is the substrate. A rather poor polish and HF contained in the etchant produced the rough surface shown. It is assumed that the breaks in the film are due to lift off because of poor film to substrate adhesion. We have found this problem to be prevalent in Nb films deposited at $< 300^{\circ}\text{C}$ substrate temperature onto Pyrex.

When the substrate temperature is raised to about 500°C and the substrate material changed to fused silica substantial improvements in etched pattern quality result as shown in Fig. 23(a). In this case the photograph was taken using reflected light so that the bright area represents the coil. Fig. 23(b) is a high magnification (1250x) photo of a single line showing the regularity of the film edges. The entire coil is shown in Fig. 24 and consists of 71 turns, with center of turn to center of turn spacing of about $72\text{ }\mu\text{m}$, turn crosssection of $35\text{ }\mu\text{m}$, turn crosssection of $35\text{ }\mu\text{m}$ by $0.5\text{ }\mu\text{m}$, outer diameter of 3.18 cm and inner diameter of 2.1 cm. The total length of conductor was estimated at 566 cm. Some preliminary electrical measurements were made on this coil and are shown in Table IX.

We are presently preparing etched patterns for the entire filter and plan to make characteristic measurements of the assembly.

a.

b.

Fig. 22 Examples of etching defects occurred in the Nb film coil on Pyrex substrate.

a.

b.

Fig. 23 Improved etching pattern of Nb film coil on fused silica substrate.

Fig. 24 Perfectly etched Nb film coil.

TABLE IX

D1 Coil Performance Data

Room Temperature Resistance	30.9 K Ω
77K "	8.0 K Ω
10K "	3.0 K Ω
R_{300}/R_{10}	10.3
Transition Temperature	9.3 K
Inductance at 4.2 K	0.21 mH

V. FILM PREPARATION FACILITIES

All superconducting films used in this program will be prepared by dc magnetron sputtering. The system employed in the early phases of the effort is an already existing facility consisting of a diffusion pumped liquid-nitrogen trapped, stainless steel vacuum chamber capable of attaining a base pressure in the 10^{-8} torr range. The chamber contains a single Varian 5 inch S-gun sputtering assembly mounted on the top plate of the chamber so as to sputter downward onto a substrate stage which can be either heated or cooled. Chromel-Alumel thermocouples monitor substrate temperatures while sputtering pressure is measured using a wide range ionization gauge or absolute capacitance manometer. 99.9999% pure Argon is used as the sputtering gas with sputtering pressures ranging from 3 to 100 mtorr. All sputtering is done under conditions of continuous Ar flow using the diffusion pump at a throttled pumping speed of 70ℓ/sec. Impurity gas content can be monitored during sputtering by means of a differentially pumped quadrupole mass spectrometer. This system has been successfully used to make very high quality superconducting films of Nb, Nb₃Sn, Nb₃Ge and NbN.

We are presently completing the design and construction of a new sputtering system consisting of a 24 inch diameter stainless steel chamber equipped with two S-guns mounted to allow upward sputtering and sequential film deposition without breaking vacuum. The chamber is being made at ANL and will be shipped to Varian for mating to a Model 3118 pumping module with two S-guns mounted on a 24" diameter base plate. The system is expected back at ANL in early October, 1978 and should be functional for film deposition by November 1, 1978. All the Nb₃Sn filter elements will be made in this system.

REFERENCES

1. K. Siegel, R. Domchick and F. R. Arams, Proc. Symposium on the Physics of Superconducting Devices, 1967, Charlottesville, VA.
2. C. C. Koch, J. O. Scarbrough, and D. M. Kroeger, Phys. Rev. B, 9, 888 (1974).
3. R. T. Kampwirth, J. W. Hafstrom, and C. T. Wu, IEEE Trans. Magnetics Mag. 13 (1), 315 (1977).
4. R. T. Kampwirth, C. T. Wu and J. W. Hafstrom, Adv. Cryogenic Eng. 24, 465 (1978).

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