

### ISTITUTO NAZIONALE di FISICA NUCLEARE

and

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At a given angular frequency  $\omega$ , the surface impedance  $Z_n$  for a normal metal, in the normal regime, can be written as



where  $\sigma_n = 1 / \rho_n$  is the dc conductivity at the working temperature;  $\delta$  is the skin depth

**Extension to Superconductors:** 

 $\sigma_1 - i \sigma_2$  in place of  $\sigma_n$ 

As derived by Nam, for T < Tc / 2

R<sub>s</sub> can be approximated by



In the framework of the BCS theory extension at finite frequencies, Mattis and Bardeen found, for  $\omega < 2 \Delta$ , the two following integral relations for the complex conductivity of a superconductor:

$$\frac{\sigma_{1}}{\sigma_{n}} = \frac{2}{h'\omega} \int_{\Delta}^{\infty} (f(E) - f(E + h'\omega)) g^{+}(E) dE$$

$$\frac{\sigma_{2}}{\sigma_{n}} = \frac{1}{h'\omega} \int_{\Delta-h'\omega, -\Delta}^{\Delta} (1 - 2f(E + h'\omega)) g^{-}(E) dE$$

$$2 \Delta = s \ K T_{C},$$
$$f(E) = \frac{1}{\left(\frac{E}{K T}\right)}$$

$$g^{\pm}(E) = \frac{E^{2} + \Delta^{2} + h'\omega E}{\left(\frac{1}{2} + (E^{2} - \Delta^{2})\right)^{1/2} \left(\left(E + h'\omega\right)^{2} - \Delta^{2}\right)^{1/2}}$$

The two integrals  $\sigma 1/\sigma n$  and  $\sigma 2/\sigma n$  are easily numerically calculated.

In the normal skin effect regime, for  $h'\omega \ll 2\Delta$ they can be approximated by two analytical expressions:



#### Then, if T < Tc / 2



with A = 
$$6 \cdot 10^{-21} \left[ \frac{\Omega K^3}{m s^4} \right]^{\frac{1}{2}}$$

#### For low rf losses, a high T<sub>C</sub> value it is not sufficient

# A metallic behaviour is mandatory



Fig 1 Lines of equal R<sub>BCS</sub> at 4.2 K and 500 MHz in the bidimentional space ( $\rho_n, T_{t}$ ). Fixed the working temperature (T = 4.2 K), and the frequency (f = 500 MHz), R<sub>BCS</sub> depends only on the energy gap and on the normal state resistivity. The Tc scale is draft for the case s = 4. For comparison Nb coated cavities provide R<sub>Bts</sub> = 55 n $\Omega$ . The experimental data refer to: sputtered films of (NbTi)N at 200 C(full square); (NbTi)N at 600 C(empty square); NbN at 200 C



**Different classes of superconductors** (after Buzea and Yamashita).



Fig. 1 Crystal struc-ture of the superconduc-tor type  $Bi_2$ ( $Sr_{1-y}Ca_y$ )<sub>3</sub> Cu<sub>2</sub>O<sub>10-5</sub> deve-loped by Hoechst. Key: green = calcium (Ca), red = copper (Cu), black = oxygen (O), yellow = stron-tium (Sr), blue = bismuth (Bi).

#### PERCOLATION, SURFACE IMPEDANCE and PINNING in MgB<sub>2</sub>-, and HTS-SUPERCONDUCTORS. J. Halbritter, Forschungszentrum Karlsruhe Institut für Materialforschung Postfach 36 40 76021 Karlsruhe

#### ABSTRACT

The hindrance of electric transport by grain/island boundary resistances  $R_{bn} (\Omega cm^2)$  in distances  $a_J (\leq 1 \mu m)$  is well accepted in the normal conducting transport in granular materials. In contrast, in superconducting transport such boundaries enhance by the related critical Josephson current density  $j_{cJ}$  the critical current density by Josephson fluxons (JF). For a quantitative model the resistivity

 $\rho(T) = R_{bn}/a_J + p \ (\rho^i \ (T) + \rho^i \ (0))$ 

is fitted to observations with percolation factors p > 1 by current diverting boundaries with  $R_{bn} \gtrsim m\Omega cm^2$ . HTS show p > 1 - 10, critical current densities  $j_e(H < H_{Cl}) \simeq j_{eJ}(H)$  by JF pinning with  $j_{eJ}R_{bn} \approx 10^{-12} V\Omega m^2/R_{bn} << \Delta/e$ , rf residual losses  $R_{res} \propto \omega^2/a_J j_{eJ}^{-3/2} R_{bn}$  and hysteresis losses  $R_{hys} \propto \omega H/a_J j_{eJ}$ . In MgB<sub>2</sub> films percolation with  $p \gtrsim 2 - 50$ , with  $j_e(H < H_{e1}) \simeq j_{eJ}(H)$  and  $j_{eJ} R_{bn} \approx \Delta/e$  is found. Whereas percolation decreases conductivities via the boundary resistances  $R_{bn}$  in the normal state always, pinning of JF enhances  $j_e(H < H_{e1}) \simeq j_{eJ}(H)$  dominated in dc transport by chains of strongest links. In contrast, rf surface impedances via  $R_{res} (T, \omega) \propto \omega^2/j_{eJ}(T)^{3/2}$  and  $R_{hys} (T, H) \propto \omega H/j_{eJ}(T)$  are dominated by the weakest links and deteriorate by weak links always. But in all cases where  $\rho(T)$  deviates from Matthiessen law, RRR values are not sufficient to describe material quality, but a proper percolation analysis is needed to forcast via  $R_{bn} \propto j_{eJ}^{-n}$  ( $n \simeq 1$ -2) superconducting properties below  $H_{c1}$  in dc and rf.



### YBCO thin films on large area substrates

P.Romano, A. Vecchione,G. Keppel and V. Palmieri

DIODE Sputtering at 950 C at 1 mbar
onto sapphire, and SrTiO3
Distance cathode substrate = 10 mm





Structure of MgB<sub>2</sub> containing graphite-type B layers separated by hexagonal close-packed layers of Mg (After Buzea and Yamashita)

# And what about the problem of film degradataion due to water exposure???

Activity on MgB2 at Los Alamos Neutron Science Center

# Report by Tsuyoshi Tajima

#### **Bulk samples by HIP**

- No degradation with high-pressure water rinse
- Surface polishing with 0.1mm diamond lapping paper reduced Rs significantly



#### Giorgio Keppel, Thesis 2004, Material Science Dept, Padua University





#### **DC** Magnetron Sputtering of MgB<sub>2</sub> + Mg



Films are deposited by an in-situ process in two steps:

DC Magnetron Sputtering of MgB<sub>2</sub> and Mg targets at room temperature

Post-annealing at increasing temperature (300°C - 600°C) in Mg sputtering



#### **DC Pulsed Sputtering of MgB<sub>2</sub> + Mg Powders**

V. Palmieri, A. Calore, I.I. Kulik

Advantages: The added Mg compensates the lost one. Targets are self-made. One can immediately prepare targets out of stoichiometry or with the addition of other elements. Disadvantages: Possible leaking of Magnesium during the sputtering process.



Room temperature substrate

Substrate Temperature = 500 C

**No Postannealing** 

T-dependence of the order parameter from fit with the one-gap model. $\Delta_{dirty}(T)$  and  $\Delta_{3D}(T)$  from fit with the two-gap model. Fit to BCS curves T-dependence of the order parameter from fit with the one-gap model. $\Delta_{dirty}$ (T) and  $\Delta_{3D}$ (T) from fit with the two-gap model. Fit to BCS curves



(after G. Carapella, N. Martucciello, G. Costabile, C. Ferdeghini, V. Ferrando, and G. Grassano)

## **Gap parameters**

from specific heat and spectroscopic experiments:

Technique	$2\Delta_1(0)/k_BT_c$	$2\Delta_2(0)/k_BT_c$	$\gamma_1:\gamma_2$
specific heat	3.8	1.3	0.5:0.5
specific heat	3.9	1.3	0.5:0.5
specific heat	4.4	1.2	0.55:0.45
penetration depth	4.6	1.6	0.60:0.40
tunneling	4.5	1.9	
Raman	3.7	1.6	
point-contact	4.1	1.7	
photoemission	3.6	1.1	
band structure	4	1.3	0.53:0.47

(After A. Junod, Y. Wang, .F. Bouquet, P. Toulemonde)

# Two fluid model Three fluid model



Equivalent circuit for the admittance of a unit cube of superconductor



under

In the TI-Pb-Bi System, the mass can be held almost constant and the variation of  $T_{C}$  with e/a can be studied in a continuous fashion even up to 9 K



Variation of Transition Temperature with electrons-to-atom ratio in the Tl-Pb-Bi alloy family. *After Dynes and Rowell, Phys. Rev. B* 11,1884 (1975) **PdH** is a very famous material for the reversal isotope effect and it has low resistivity

And Hydrides of Pd-Ag and Pd-Cu display Tc up to 15K!!!

#### Critical Temperature of compounds with NaCl structure

BA	Sc	Y	La	Ti	Zr	Hf	V	Nb	Ta	Cr	Мо	W	Re
В	$\{h_i\}_{i \in \mathbb{N}}$	Sec.		200	3.4	3.1						-	
С	<1.38	<1.38		3.42	<0.3	<1.20	0.03 3.2*	12	10.35		14.3	10.0	3.4
N	<1.38	<1.4	1.35	5.49	10.7	8.83	8.5	17.3	6.5	<1.28	5.0	<1.38	1
Р	6.61		<1.68							$\frac{1}{2} = -\frac{1}{2} \left( \frac{1}{2} \right)^{\frac{1}{2}}$			
Sb		<1.02	<1.02						- 		$2^{-1}$		
0	12	121	22	2.0			<0.3	1.39	2.1			1.18	6 1 <b>5 19</b> 4 1 7 4
S	<0.33	1.9	0.87		3.3		$\mathcal{X} = \mathcal{X}$				2.1	7	23
Se	<0.33	2.5	1.02				$(\cdot, \cdot)$			1.1		1.1	
Te		2.05	1.48						•				

Critical temperature vs composition for nitride and carbide addition to NbN





A.Nigro, G.Nobile, <u>V.Palmieri</u>, R.Vaglio, "PROPERTIES OF NIOBIUM-TITANIUM NITRIDE SUPERCONDUCTING THIN FILMS", Adv. Cryog. Eng. Mat., vol. 34, (1988) 813





### Surface resistance of a (Nb<sub>0.55</sub>Ti<sub>0.45</sub>)N/Cu film (4 GHz)







Nontransition elements	T <sub>c</sub> (K)	Transition elements	T <sub>c</sub> (K)
Ti <sub>3</sub> Sb	6.5	Ti <sub>3</sub> Ir	4.2
Zr <sub>so</sub> Sn <sub>20</sub> <sup>a</sup>	0.92	Ti <sub>3</sub> Pt	0.5
Zr-Pb	0.76	Zr <sub>3</sub> Au	0.9
Zr~3Bib	3.4	V20Re21	8.4
V–Al <sup>c</sup>	14	V <sub>50</sub> Os <sub>50</sub>	5.7
V <sub>3</sub> Ga	15.9	V <sub>65</sub> Rh <sub>35</sub>	$\simeq 1$
V,Si	17.0	V <sub>63</sub> Ir <sub>37</sub>	1.7
V <sub>~3</sub> Ge	6	V <sub>~3</sub> Pd VPt	0.08
V_3Ge	3.8	V <sub>3</sub> rt V <sub>2</sub> Au	3
~ ~ 79 <sup>-244</sup> ~ 21	0.0	, 76° 1024	
V <sub>77</sub> As <sub>23</sub>	0.2	Nb <sub>75</sub> Os <sub>25</sub>	1.0
V <sub>76</sub> SD <sub>24</sub>	0.8	ND <sub>75</sub> Kn <sub>25</sub>	2.0
Nb <sub>3</sub> Al	19.1	Nb.Pt	11
Nb <sub>3</sub> Ga	20.7	Nb Au	11.5
Nb <sub>~3</sub> In <sup>b</sup>	9.2	~, T- D:	0.4
$ND_{82}S1_{18}^{a}$	4.4	$Ta_{85}Pt_{15}$	0.4
Nb–Si°	11-17	$1a_{\sim 80}Au_{20}$	0.55
Nb-Ge <sup>a</sup>	17	Cr <sub>72</sub> Ru <sub>28</sub>	3.4
Nb-Ge <sup>c</sup>	23	Cr <sub>73</sub> Os <sub>27</sub>	4.7
Nb <sub>3</sub> Sn	18	$Cr_{78}Rh_{22}$	0.07
ND-SD Nb Bib	2	$Cr_{82}Ir_{18}$	0.75
IND~3DI	5	Mo40Tc60	13.4
Ta <sub>~3</sub> Ge <sup>c</sup>	8	Mo <sub>~65</sub> Re <sub>~35</sub> °	$\simeq 15$
Ta <sub>~3</sub> Sn	8.3	Mo <sub>75</sub> Os <sub>25</sub>	13.1
	0.7	MO <sub>78</sub> Ir <sub>22</sub> Mo Pt	8.5
Mo <sub>3</sub> Al	0.58	1VIO <sub>82</sub> F1 <sub>18</sub>	4.0
Mo <sub>3</sub> Ga	0.76	$W_{\sim 60}Re_{\sim 40}c$	11
Mo <sub>77</sub> Si <sub>23</sub>	1.7		
$Mo_{77}Ge_{23}$	1.8		

a Rapid quenching b High-pressure synthesis c Film deposition techniques



Nb<sub>3</sub>Sn 1.5GHz cavity made at Wuppertal by Sn vapour phase diffusion



comparison to pure Nb at 4.2 K and 2 K from CEBAF.





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# *Tc vs Si content for sputtered films before and after in situ post-annealing in SiH4 atmosphere*



Y. Zhang, V. Palmieri, W. Venturini, F. Stivanello, R. Preciso, LNL-INFN (REP) 157/2000

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For Nb: several precursors i.e. Niobium pentakis(dimethilammide) For Sn: Bu3SnH

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### Synthesis of Niobium Pentakis(dimethilammide)

The reaction happens in two different steps:

First the Me<sub>2</sub>NH 50 mL is bubbled for around 90 minutes in LiBu:

 $Me_2NH + LiBu \rightarrow LiNMe_2 + BuH$ 

Than the butane is evaporated and the product obtained is suspended in pentane and treated by NbCl<sub>5</sub>

pentane

# $NbCl_{5} + 5LiNMe_{2} \rightarrow Nb(NMe_{2})_{5} + 5LiCl$





The Ammide is a brrownish powder that sublimes at 130°C

#### RBS del deposito 1 da CpNbMe<sub>4</sub>



### A possible source of oxygen contamination



#### Molybdenum-Rhenium

Most commonly known as **Moly-Rhenium**, and used extensively throughout many industries -from medicine to defense and pure research to production welding, this material is a less costly alternative to pure rhenium.

Possessing excellent thermal and mechanical properties, **Moly-Rhenium** is used as welding wire, wires for numerous medical applications, components and parts for the aerospace and defense industries, and grids for electronic applications.

Density, g/cm <sup>3</sup>	13.52
Melting Point, °C	2550
Thermal Conductivity, W/m at 20°C	36.8
Linear Coefficient of Thermal Expansion, µm/m·K from 20-1000°C	5.7
Ductile Brittle Transition Temperature (DBBT), °C	(-273)-(-173)
Critical Superconducting temperature, K	10.9
Electrical Resistivity, μΩ·m at 20°C	0.220
Elastic Modulus in Tension, GPa	373









(After Yasaitis and Rose, 1975)



Nb<sub>3</sub>Sn 1.5GHz cavity made at Wuppertal by Sn vapour phase diffusion



comparison to pure Nb at 4.2 K and 2 K from CEBAF.



Curve A - Films sputtered at ~ 500 Å/min onto 1000 °C substrates Curve B – Films sputtered at ~ 1000 Å/min onto 1200 °C substrates Curve C – Bulk Mo-Re samples

From Blaugher et al

#### Mo<sub>38</sub>Re<sub>62</sub> (after Testardi)



(The author communicates that the Temperature was 150 C lower than reported in the picture)



Fig. 3. T's of Mo-Re alloy samples all of the approximate composition  $Mo_{40}Re_{60}$ . A-Bulk sample<sup>4</sup>; B, C, D, E- thin films sputtered at ~ 1000 Å/min onto 600°C, 750°C, 1150°C, and 1200°C substrates, respectively.

 $\mathbf{R}_{\mathrm{S}} = \mathbf{R}_{\mathrm{S}} \; (\mathrm{e}^{-\mathrm{sTc}/2\mathrm{T}})$ 

Measured Parameters of V1-B, VII-B Superconductors

Compound	$\gamma(mJ/g-atom-K^2)$	$\beta(mJ/g-atom-K^4)$	δ(mJ/g-atom-K <sup>6</sup> )	θ <sub>D</sub> (K)	24/kTc	T <sub>c</sub> onset	Struct.
Mo0.6 <sup>Re</sup> 0.4	4.55	.05639	.0001535	325 340	5.0	12.0 12.6	bcc bcc
	4.44	.0481	0	342	3.4	6.0	tetr.
<sup>Mo</sup> 0.4 <sup>Re</sup> 0.6	3.80	-	a.a. <sup>Ra</sup> ata	355		6.49	tetr.
<sup>Mo</sup> 0.42 <sup>Re</sup> 0.58	3.31	-		351	2.8	6.35	tetr.
<sup>Mo</sup> 0.23 <sup>Re</sup> 0.77	3.8	-	- 66	272	3.0	9.25	A-12
W <sub>0.65</sub> <sup>Re</sup> 0.35	2.49	.06599	0	309	3.1	6.75	bcc
W <sub>0.80</sub> <sup>Re</sup> 0.20	2.2		-	359	17	-	bcc
W <sub>0.75</sub> <sup>Re</sup> 0.25	2.3	-		351		-	bcc
W <sub>0.59</sub> <sup>Re</sup> 0.50	2.69	-		327	3.3	5.12	tetr.
Mo <sub>0.18</sub> Tc <sub>0.82</sub>	8.	.03396	.0002317	385	3.6	13.7	hcp





A.Andreone, A.Barone, A.Di Chiara, G.Mascolo, <u>V.Palmieri</u>, G.Peluso, U.Scotti, "Mo-Re Superconducting Thin Films by Single Target Magnetron Sputtering", **IEEE Trans. Mag., 25, 2, (1989) 1972** 



# A 6 GHz seamless cavity

## A cheap way to fabricate samples for RF properties from Nb retails 15 minutes of fabrication time, including flanges,





