

### ISTITUTO NAZIONALE di FISICA NUCLEARE

and

Padua University – Material Science Departement, ITALY

Argonne National Laboratories,IL, Sept 21 2004

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.





Silvia Deambrosis, Thesis 2004, Material Science Dept, Padua University







# *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

Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



Francesco Todescato, Thesis 2004, Material Science Dept, Padua University



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

Francesco Todescato, Thesis 2004, Material Science Dept, Padua University

### 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

![](_page_55_Figure_1.jpeg)

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

![](_page_56_Figure_0.jpeg)

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)

![](_page_57_Figure_1.jpeg)

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

![](_page_58_Figure_0.jpeg)

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

![](_page_60_Figure_0.jpeg)

![](_page_61_Figure_0.jpeg)

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** 

![](_page_62_Figure_0.jpeg)

# A 6 GHz seamless cavity

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

![](_page_63_Picture_2.jpeg)

![](_page_64_Figure_0.jpeg)

![](_page_65_Figure_0.jpeg)