

Physical Properties and Testing

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Outline

• Introduction

Physical Properties of materials in accelerator applications.

 $\,\circ\,$ University of Zaragoza labs

• Electrical Properties of Materials:

- \circ R(T), RRR, T_c
- Testing Methods and standards
- \circ Examples

• Thermal Properties:

- \circ c_P(*T*,*H*), *k*(*T*)
- \circ Testing methods
- Examples: Nitronic 50 austenitic steel

• Magnetic Properties,

- \circ M(H), $\mu_r(H)$
- \circ Hysteresis main parameters
- $\,\circ\,$ Testing Methods and standards
- Examples: Non magnetic alloys, magnetic steel, manufacturing





Physical Properties of materials at extreme conditions

High Radiation dose (accumulative damage) Very low T (1.9K superfluid LHe) High T gradients High Magnetic Fields (8.3 T $\approx 2 \cdot 10^5 B_{\text{Farth}}$) High vacuum

> Robust carbon-fibre composites and non-robust but highabsorption metallic materials (W) are used

cleaning insertion

cold

anertu

device













arc(s)





Physical Properties of materials at extreme conditions

Electrical Properties of Materials: -Resistivity, electrical transport

Thermal Properties:

- Heat capacity
- Thermal conductivity

Magnetic Properties





Cryogenic Temperatures: *T* < 10 K High Magnetic Fields: *B* > 2 T







Physical Properties of materials at extreme conditions: low *T*, high *H* Physical Properties Measurement System: PPMS-14T

1.9 K – 400 K 14T Magnetic field

350 K – 0.35 K Electrical Properties of Materials •Heat Capacity

Thermal Properties: Magnetic Properties



PPMS with ³He refrigerator









Sample stage







UZ Research Labs

SQUID Magnetometer MPMS3



EQC2021-007030-P. *Magnetometro SQUID Sub-K*. Funding Agency: European Union, NextGenerationEU funds. EQC2021 call. 854 k€.

- Options for Standard *T* : 1.8 400 K
 - VSM/DC mode
 - AC measurement system
 - 0.1 Hz 1000 Hz
 - Rotator single crystal
 - Fiber optic Magneto-optic
 - Pressure Cell





Introduction Physical Properties of materials at extreme conditions: low *T*, high *H*



UZ Research Labs

Sub-K SQUID Magnetometer MPMS3

Extending the Temperature Range

Helium-3 Refrigerator2 K – 0.4K



Electrical Properties of Materials





Classical view

The electrical properties of a solid material are a consequence of its electron band structure





Electrical Properties of Materials: metals

Electron





level defects. Scattering of the electrons reduces the mobility and

conductivity.

$$\rho_t = \rho_0 + aT$$



Increase with temperature

Increase in thermal vibrations and other lattice irregularities (e.g., vacancies): electron-scattering centers.

Constant at very low temperature

the mean free path is dominated by impurities or defects in the material





Electrical Properties of Materials



'sweet spot' R ~ 1 Ohm -1 MOhm











R=V/I





PC-RES-P

Electrical Properties of Materials



Measure of the superconducting transition $T_c(T,H,I)$



2D Mo/Au bilayers Transition Edge Sensors (TESs)





Electrical Properties of Materials: metals

 ρ_{300K}

 ho_{0K}





It serves as a rough index of the purity and overall quality of a sample



RRR =

LHC RF cavities operate at 4.5 K

Pure niobium in its superconducting state ($T_c = 9.2K$) is used for highquality-factor resonant cavities for particle accelerators.

Interstitial impurities (O, N, C, H) have major influence on the thermal conductivity of niobium, damaging cavity performance. The total impurities content can be determined by measuring the RRR.



Thermal Properties

Temperature Heat Management



Heat Capacity: Specific Heat – $C_{\rm P}$

- Information extracted, models
- Adiabatic, semiadiabatic

Thermal Conductance, *K*: Thermal conductivity *k*

- Information extracted, models
- Steady-state, Dynamic



RADIATION

CONVECTION

HEAT

ENERGY

Experimental complications and constraints

- Heat Flow control
- Heat radiation
- Small Temperature differentials measurements





Specific Heat: Most fundamental thermodynamic property

Related to the internal energy of a system Contains information about all the degrees of freedom

$$C = C_{ph} + C_m + C_e + C_{hf} = C_{ph} + C_m + \gamma T + \frac{\alpha}{T^2}$$

Solid as an isotropic continuum médium

Lattice Debye Model

Lattice specific heat, determined by the normal modes of vibrations available to its structure



 $x = \hbar \omega / k_B T$ $T_{\rm D} = \hbar \omega_{\rm D} / k_{\rm B}$

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ollective motions called phonons. Credit: Sean Kellev/NIST



Low *T*, *T*<<*T*_D
$$C_P \cong \frac{12\pi^4}{5} NR \left(\frac{T}{T_D}\right)^3 = AT^3$$

High *T*, *T*>>*T*_D $C_P \cong 3NR$





 $C_P \cong C_V = C_{ph} = 9Nk_B \left(\frac{T}{T_D}\right)^3 \int_0^{x_D} dx \frac{x^4 e^x}{(e^x - 1)^2}$

Specific Heat: Standard Characterization



Low T C(T) ~ AT^3 25 8²⁰ graphit 口水布 15 C(T), C_L Characterization: Cv, Cp 10 diam ond Sensible heat, Latent Heat **Debye Temperature** 200 400 1000 600 800 $\theta_{\rm D} = T_{\rm D} = \hbar \omega_{\rm D} / k_{\rm B} = \hbar \upsilon_{\rm s} (6\pi^2 N/V)^{1/3} / k_{\rm R}$ Temperature (K) u_s velocity of sound The Debye temperature $\theta_{\rm D}$ is the temperature of a crystal's highest Latent Heat normal mode of vibration, and it First Order Phase Transitions FOPT correlates the elastic properties

(structural rigidity), thermal

specific heat

expansion, thermal conductivity, and

First Order Transitions



Figure 2.2: Temperature dependence of the heat capacity at zero field for $Mn_{0.997}Co_{0.003}As$. The value of shaded area indicates the latent heat associated to a FOPT. The stepped line represents the normal heat capacity.





Heat Capacity semi-adiabatic relaxation method

Implementation in HC option of PPMS Quantum Design^(*)









Samples: 0.1 mg – 100 mg Solid, powder, 'liquid'

Model limitation for *C* divergence (1st order transition)





 > Single pulse análisis
 > Relaxation method with small ∆T steps (infinitesimal approach)



http://sai.unizar.es/medidas-fisicas-eng/hc

Critical examination of heat capacity measurements made on a Quantum Design physical property measurement system, J.C. Lashley et al. Cryogenics, Volume 43, Issue 6, 2003, Pages 369-378, https://doi.org/10.1016/S0011-2275(03)00092-4.

Thermal Conductivity

• Thermal conductance: ratio of steady-state heat flow to temperature difference

 $K = \frac{Q_{th}}{\Lambda T} W/K$ $\kappa = K(L/A) W/m \cdot K$

Sources of phonon scattering: Defects, interactions, interfaces

Heat transfer at the nanoscale \succ Size dependent termal conductivity

Low-energy phonon structure

INS + Calculations \Leftrightarrow Experimental k

Study of processes taking place in thermal conduction

Information on the scattering of electrons and phonons



 $\kappa = 1/3 C < v > </>$







Amorphous solids $k \propto T^2$



Thermal Conductivity Techniques



1-heater 2-thermometers method Thermal Circuit







> Thermal Transport sample puck with radiation shield





Thermal Conductivity Techniques: comercial TTO from PPMS (QD)

•Temperature: 1.9 K - 390 K

•High vacuum (~10⁻⁴ torr)

Magnetic Field
 0 a ± 140 kOe for T > 20 K
 0 a 1 kOe for T < 20 K

• Range of k and sample geometries

10-150 W/mK (Needle: 10x1x1 mm³) 2-30 W/mK (Brick: 8x2x2 mm³) 0.1-1.5 W/mK (Pill: 3x5x5 mm³)

Limitations: High k: Maximum heating power 50 mW Low k : Measurement time

Lead Configuration:2-Probe4- Probe





Carbon fiber/epoxy composites







Thermal Conductivity TTO

Steady-state solution

Only for very high k samples k > 200 W/mK



$$\Delta T(t) = \frac{P_0}{K_s} \left[1 - \left(\frac{\tau_1 e^{-t/\tau_1} - \tau_2 e^{-t/\tau_2}}{\tau_1 - \tau_2} \right) \right]$$



In the asymptotic limit,

$$\Delta T(t \to \infty) = \Delta T_{\infty} = \frac{P_0}{k}$$

 P_0 is the power supplied to the sample, nominally $P_{\rm htr} = I^2 R_{\rm htr}$

Radiated Power: $P_{rad} = \sigma_T \times \left(\frac{S}{2}\right) \times \epsilon \times (T_{hot}^4 - T_{cold}^4)$ σ_T is the Stefan-Boltzmann constant S and ϵ are the surface area and infrared emissivity of the sample, respectively

$$K_S \equiv \left(\frac{(P_{\rm htr} - P_{\rm rad})}{\Delta T_{\infty}} \right) - K_{\rm shoes}$$

Finally, geometric factors can be applied to express this as the intrinsic thermal *conductivity*:

$$c_{S} = \frac{L}{A} \left[\left(\frac{(P_{htr} - P_{rad})}{\Delta T_{\infty}} - K_{shoes} \right] \right]$$

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 ΔT_{∞}



Physical Properties of Nitronic 50 ITER

Nitronic[®] 50 High Strength high N and Mo austenitic stainless steel **Non-magnetic, with low μ even at cryogenic T's**.

ITER project (International Thermonuclear Experimental Reactor). Central solenoid compression structure



Central solenoid: the strongest of all ITER magnets Maximum field of 13 T

https://www.iter.org/newsline/-

/3233#:~:text=The%20ITER%20central%20solenoid%20is,a%20pre%2Dcompression%20support%20structure.

Collaboration University of Zaragoza - Engineering Department at CERN. A. Arauzo and Stefano Sgobba.





S. Sgobba, S. A. E. Langeslag, A. Arauzo, P. Roussel and P. Libeyre, "Physical Properties of a High-Strength Austenitic Stainless Steel for the Precompression Structure of the ITER Central Solenoid," in IEEE Transactions on Applied Superconductivity, vol. 26, no. 4, pp. 1-4, June 2016, Art no. 7000104, doi: 10.1109/TASC.2016.2535388.

The tie plates **operating at 4 K** can be manufactured:

- a) from single forgings ("singlepiece forging" tie plate), involving blanks of more than 15 m length
- b) from shorter forgings welded together

We have studied samples belonging to two different series in three different positions:

Single Piece Forged, SPF:

SPF-TOP SPF-MIDDLE SPF-BOTTOM

Welded Solution, WS:

WS-TOP WS-MIDDLE WS-BOTTOM













Physical characterization comprises the following measurements:

- •Measurements of Thermal Conductivity and Electrical Resistivity between 293K and 4K.
- •Measurements of Specific Heat between 293K and 4K.
- •Measurements of Magnetization and Magnetic Permeability 293K 4K at H = 7950 A/m (H=100 Oe).
- •Hysteresis cycle at 295K and 4.2K up to 7162 kA/m (*H*=90000 Oe).

Thermal conductivity and specific heat capacity are significant in predicting the quench behavior and cooling process of the large-scale superconducting magnet.











Heat Capacity:

specific heat $c_{P}(T)$

Physical properties of NITRONIC 50[®] RT Thermal capacity, c_p : 0.5 kJ • kg⁻¹ • K⁻¹





Thermal Conductivity: k

k(*T*)

Physical properties of NITRONIC 50[®] Thermal conductivity, k: 15 W \bullet m⁻¹ \bullet K⁻¹









Electrical Resistivity: $\rho(T)$

Physical properties of NITRONIC 50[®] Electrical Resistivity, 21 °C 82 $\mu\Omega$ •cm

Super A	Alloy	Nitronic	50

Carbon	0.06 max
Chromium	20.5 - 23.5
Iron	Balance
Manganese	4 - 6
Molybdenum	1.5 - 3
Nickel	11.5 - 13.5
Niobium	0.1 - 0.3
Nitrogen	0.2 - 0.4
Phosphorus	0.04 max
Silicon	1 max
Sulphur	0.03 max
Vanadium	0.1 - 0.3









Magnetic Properties of Materials Fundamentals

• Every material is magnetic: shows a kind of magnetic behavio



Commonly: magnetic = magnet

- A magnetometer measures the magnetic moment, the amount of magnetism in a sample.
- By studying the variation of the magnetization with temperature or the magnetic field intensity we may determine the type of magnetism of a material.
 - *M*(*H*), *M*(*T*)

Magnetic Properties of Materials

- Characteristic Magnitudes
 - Magnetic Field, *H* [A/m]
 - Magnetization, $M [A/m] \rightarrow M = \chi H$
 - Flux density, $B[T] \rightarrow B = \mu H$
 - Susceptibility, χ
 - Permeability, μ , $\mu_r = \mu/\mu_0$

Related by:

$$\begin{split} B &= \mu_0 H + \mu_0 M = \mu_0 \ (1 + \chi) \ H = \mu H \\ \mu &= \mu_0 \ (1 + \chi) \end{split}$$

Magnetization, *M*(*H*,*T*) Static magnetic properties

Types of magnetic behavior

- Diamagnetic
- Paramagnetic
- Magnetic order
 - Ferromagnetics
 - Antiferromagnetics
 - Ferrimagnetics
- Superconductors
- Superparamagnetism (NP's)







Magnetic Properties of Materials





Paramagnetic Ions

	Table 4.7. The so lons. m_{eff} is in units of μ_B													
3d*		s	L	J	8	$m_{eff} = \frac{m_{eff} - m_{eff}}{g\sqrt{J(J+1)}}$	$m_{eff} = g\sqrt{S(S+1)}$	~J						
1	Ti ³⁺ , V ⁴⁺	1 2	2	1/2	4/5	1.55	1.73	1.7						
2	Ti ²⁺ , V ³⁺	1	3	2	2	1.63	2.83	2.8						
3	V ²⁺ , Cr ³⁺	3	3	3	2	0.78	3.87	3.8						
4	Cr ²⁺ , Mn ³⁺	2	2	0			4.90	4.9						
5	Mn ²⁺ , Fe ³⁺	2	0	2	2	5.92	5.92	5.9						
6	Fe ²⁺ , Co ³⁺	2	2	4	3	6.71	4.90	5.4						
7	Co ²⁺ , Ni ³⁺	3	3	2	4	6.63	3.87	4.8						
8	Ni ³⁺	1	3	4	24	5.59	2.83	3.2						
9	Cu ²⁺	1	2	2	<u>6</u>	3.55	1.73	1.9						

Table 4.6. The 4 f ions. The paramagnetic moment m_{df} and the saturation moment m_0 are in units of μ_B												
$4f^{*}$		S	L	Ą	8	$m_0 = gJ$	$m_{eff} = g\sqrt{J(J+1)}$	m_{q}^{ap}				
1	Ce ³⁺	1/2	3	5	47	2.14	2.54	2.5				
2	Pr ³⁺	1	5	4	4 3	3.20	3.58	3.5				
3	Nd ³⁺	3	6	2	8	3.27	3.52	3.4				
4	Pm ³⁺	2	6	4	3	2.40	2.68					
5	Sm ³⁺	5	5	5	27	0.71	0.85	1.7				
6	Eu ³⁺	3	3	0	0	0	0	3.4				
7	Gd ³⁺	7	0	7	2	7.0	7.94	8.9				
8	Tb ³⁺	3	3	6	3	9.0	9.72	9.8				
9	Dy ³⁺	5	5	15 2	4	10.0	10.65	10.6				
10	Ho ³⁺	2	6	8	<u>5</u> 4	10.0	10.61	10.4				
11	Er ³⁺	1/2	6	15 2	6 5	9.0	9.58	9.5				
12	Tm ³⁺	1	5	6	7	7.0	7.56	7.6				
13	Yb ³⁺	1/2	3	7	<u>8</u> 7	4.0	4.53	4.5				

		⁸⁹ Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	¹⁰¹ Md	102 No	103 Lr	
		57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dv	67 Ho	68 Fr	⁶⁹ Tm	70 Yb	71 Lu	
⁸⁷ Fr	Ra		¹⁰⁴ Rf	105 Ha	106 Sg	¹⁰⁷ Bh	¹⁰⁸ Hs	¹⁰⁹ Mt	110 Ds	¹¹¹ Rg	¹¹² Cn	¹¹³ Nh	¹¹⁴ Fl	¹¹⁵ Mc	116 Lv	¹¹⁷ Ts	¹¹⁸ Og
55 Cs	⁵⁶ Ba		72 Hf	⁷³ Та	⁷⁴ W	75 Re	76 Os	⁷⁷ lr	78 Pt	79 Au	80 Hg	⁸¹ Tl	⁸² Pb	⁸³ Bi	⁸⁴ Po	⁸⁵ At	⁸⁶ Rn
³⁷ Rb	³⁸ Sr	³⁹ Y	40 Zr	⁴¹ Nb	42 Mo	43 Tc	⁴⁴ Ru	⁴⁵ Rh	⁴⁶ Pd	47 Ag	⁴⁸ Cd	49 In	⁵⁰ Sn	⁵¹ Sb	52 Te	53	⁵⁴ Xe
¹⁹ K	20 Ca	²¹ Sc ²² Ti ²³ V ²⁴ Cr ²⁵ Mn ²⁶ Fe ²⁷ Co ²⁸ Ni ²⁹ Cu ³⁰ Zn ³¹ Ga ³² Ge ³³ As ³⁴ Se ³⁵ Br									³⁵ Br	³⁶ Kr					
Na ¹¹	¹² Mg	a	ntiferro aramag	magnet netic	ic	superconducting (more conditions) no data							¹⁴ Si	¹⁵ P	¹⁶ S	¹⁷ Cl	¹⁸ Ar
³ Li	⁴ Be	fe	erromaį erromaį	gnetic gnetic (le	ow T)	diamagnetic superconducting (low T)							⁶ C	⁷ N	⁸ 0	9 F	Ne
́Н		Magnetic properties of elements														² He	

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Magnetic Properties of Materials Ferromagnetism

- •FM is the strongest magnetism in materials
- •Characteristic curves *M*(*H*) and *M*(*T*)
 - *M*(*H*) is not lineal and irreversible-> Hysteresis
 - Characteristic Parameters, $M_{\rm S}, M_{\rm rem}, H_{\rm c}, \mu$







Magnetic Properties of Materials Antiferromagnetism

•In an AF material neighbour magnetic moments align antiparallely canceling each other :

- Very low M values
- Linear M(H) similar to a paramagnet
- X is a valid property

Ferrimagnetism

•In a Ferrimagnet, neighbour mometns are alligned antiparallely same as in a AF, but they do not cancell each other: Similar behavior to a FM







Magnetic Properties of Materials



Ferromagnetism: Magnetic domains, anysotropy



Magnetic Anisotropy

- Crystalline \Rightarrow SO and Crystal field coupling
- Magnetoelastic \Rightarrow by deformation
- Shape ⇒ Demagnetizing field







Grain structure of a high-density magnetic storage); the arrows in some of the grains indicate the texture, or the direction of easy magnetization.



Magnetic Properties of Materials



ASTM Standards American society for testing and materials standard ASTM A342, method 5 Standard Test methods for permeability of weakly magnetic materials

 μ_{r} < 6.0

IEC International Electrotechnical Commission) INTERNATIONAL STANDARD IEC 60404-5 Magnetic materials – Part 5: Permanent magnet (magnetically hard) materials – Methods of measurement of magnetic properties

Tumanski, S. (2011). Handbook of Magnetic Measurements (1st ed.). CRC Press. https://doi.org/10.1201/b10979



Magnetic Properties of materials: NITRONIC 50



Magnetic permeability:

Μ(*T,H***), μ(***T,H***)**

Magnetic susceptibility of this austenitic Fe-Cr-Ni alloy peaks at approximately 53.5 K (AF transition)

Physical properties of NITRONIC 50[®] Limited magnetic permeability $\mu \le 1.03$ Cryogenic temperatures

Delta ferrite (bcc **FM**) may form during solidification of steels and welds, remaining stable at all T





Non-magnetic Tungsten heavy Alloy material.

ρ = 17 - 19g/cm₃

Inermet[®] 180 Tungsten Heavy Alloy (IT180) tungsten content (> 90%) and a NiCu binder phase

High-density alloys used in the shielding of the ITER (International Thermonuclear Experimental Reactor) fusion reactor.



Tungsten alloy is used across a range of industries as a shield against gamma and x-ray radiation or as a collimator to focus radiation beams onto a target. With a density 60% greater than lead, it offers superior attenuation characteristics and allows the thickness of shielding to be reduced

μ_r (T,H)

Magnetic Properties of Materials



non-magnetic copper alloys

Permeability at a fixed temperature of 1.9K as a function of magnetic field up to H = 11,141 kA/m (14 T).



 $\begin{array}{l} \text{CuAl7Si} \Rightarrow \text{magnetic impurities} \\ \text{CuSn8P} \Rightarrow \text{Diamagnetic} \\ \text{CuSn5} \Rightarrow \text{Diamagnetic} \end{array}$

CERN Project FCC Future Circular Collider 16T dipole magnets









magnetic steels

FeNi36 alloy magnetic steel (INVAR[®]) 300-TG-180 microalloyed magnetic steel 500-TG-179 microalloyed magnetic steel 700-TG-178 microalloyed magnetic steel

CERN Project FCC Future Circular Collider 16T dipole magnets



Magnetic polarization saturation

T = 1.9 K *B* = 14T



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Nanocrystalline Soft magnetic materials FeSi alloys **FINEMET**[®] Nanocrystalline Ribbons of FeSiNbCu Amorphous Ribbons of FeSiB

Curie Temperature



Annealing process of the material

M1(Temp) is the first heating magnetization curve

M2(Temp) successive heating curves

The heating process causes a nanocrystallization of the amorphous alloy

Ferro-paramagnetic transition of the amorphous alloy Curie Temperature from the initial crystalline phase (Fe and Si) Curie Temperature from the main crystalline phase (Fe and Si)

Typical behavior of amorphous magnetic metallic alloys based on the FeSiB system





Non magnetic stainless steel

Austenitic stainless steel sample with partial copper plating and a longitudinal laser weld along the center

P506 AUSTENITIC STAINLESS STEEL SAMPLE



The low field range analysis shows:

- small magnetic remanence
- upturn of permeability









Non magnetic stainless steel

AISI 316

EN 1.4401 – X5CrNiMo17-12-2 (AISI 316)

EDM (Electrical Discharge Machined) cutting process affects the magnetic properties of the sample surface.



magnetic permeability of non-magnetic AISI 216 steels at room temperature as a function of the magnetic field up to 50 kOe





SEM (CERN) image of a FIB section of the surface of one of the samples where the affected surface is visible

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Summary



- Materials for particle accelerators are working under extreme physical conditions: *T*, *H*, *P*, radiation
- > Physical Properties of materials change with *T*, *H*.
 - R(T)c_P(T,H), k(T) $\mu_r(T,H)$
- Materials physical characterization at low T and/or high magnetic fields are required to asses requirements (technical specifications) for the final application.
- Advanced study of materials in extreme conditions provides additional fundamental information (phase transitions and stability, thermodynamics, defects, processing of materials, ...)









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Thank you for your attention



