King Saud University
College of Sciences
Physics and astronomy department

Laboratory of Solid state Physics

491 PHYS

Contents

Dielectric constant

Pre Lab Questions:

1- Discuss what happens when a dielectric material is inserted between the plates of a capacitor, explaining the terms: Dielectric constant, polarization, electric permittivity and electric susceptibility, and deriving their relations with each other's in solid?

2- What is Curie-Weiss law?

3- What are the electrical and related structural properties of $BaTiO_3$ on either side of the inversion temperature?

4- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➢ Study the change of electrical properties of the dielectric material Barium titanate ($BaTiO_3$) with temperature.

➢ Find out Curie's Temperature.

➢ Calculate Curie's Constant.

To achieve these aims study the change of the capacitor with temperature for our sample then do the calculation and charts that you are need.

In your REPORT write down everything you used or found for this experiment. About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

DIELECTRIC AND CURIE TEMPERATURE MEASUREMENT OF FERROELECTRIC CERAMICS

INTRODUCTION

Research in the area of Ferroelectrics is driven by the market potential of next generation memories and transducers. Thin films of ferroelectrics and dielectrics are rapidly emerging in the field of MEMS applications. Ultrasonic micro-motors utilizing PZT thin films and pyroelectric sensors using micro-machined structures have been fabricated. MEMS are finding growing aplication in accelerometers for air bag deployment in cars, micro-motors and pumps, micro heart valves, which have reached the commercial level of exploitation in compact medical, automotive, and space applications. Extremely sensitive sensors and actuators based on thin film and bulk will revolutionize every walk of our life with Hi-Tech gadgets based on ferroelectrics. Wide spread use of such sensors and actuators have made Hubble telescope a great success story. New bulk ferroelectric and their composites are the key components for the defence of our air space, the long coastline and deep oceans.

The quest of human beings for developing better and more efficient materials is never ending. Material Scinece has played a vital role in the development of society. Characterization is an important step in the development of different types of new materials. This experiment is aimed to expose the young students to Dielectric and Curie Temperature Measurement techinque for Ferroelectric Ceramics.

Dielectric or electrical insulating materials are understood as the materials in which electrostatic fileds can persist for a long time. These materials offer a very high resisitance to the passage of electric current under the action of the applied *direct-current* voltage and therefore sharply differ in their basic electrical properties from conductive materials. Layers of such substances are commonly inserted into capacitors to improve their performance, and the term *dielectric* refers specifically to this application.

The use of a dielectric in a capacitor presents several advantages. The simplest of these is that the conducting plates can be placed very close to one another without risk of contact. Also, if subjected to a very high electric field, any substance will ionize and become a conductor. Dielectrics are more resistant to ionization than air, so a capacitor containing a dielectric can be subjected to a higher voltage. Also, dielectrics increase the capacitance of the capacitor. An electric field polarizes the molecules of the dielectric (Figure-1), producing concentrations of charge on its surfaces that create an electric field opposed (antiparallel) to that of the capacitor. Thus, a given amount of charge produces a weaker field between the plates than it would without the dielectric, which reduces the electric potential. Considered in reverse, this argument means that, with a dielectric, a given electric potential causes the capacitor to accumulate a larger charge.
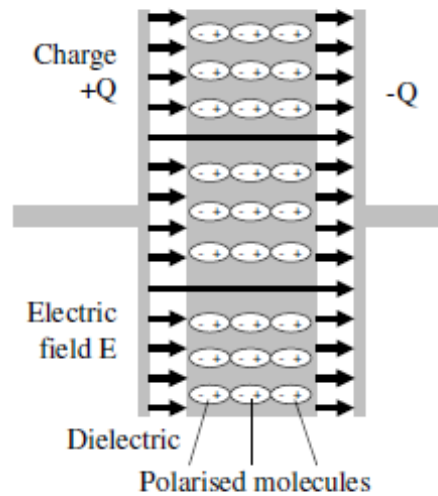
Figure-1

The electrons in the molecules shift toward the positively charged left plate. The molecules then create a leftward electric field that partially annuls the field created by the plates. (The air gap is shown for clarity; in a real capacitor, the dielectric is in direct contact with the plates.)

Perovskite Structure

Perovskite is a family name of a group of materials and the mineral name of calcium titanate ($CaTiO_3$) having a structure of the type ABO_3. Many piezoelectric (including ferroelectric) ceramics such as Barium Titanate ($BaTiO_3$), Lead Titanate ($PbTiO_3$), Lead Zirconate Titanate (PZT), Lead Lanthanum Zirconate Titanate (PLZT), Lead Magnesium Niobate (PMN), Potassium Niobate ($KNbO_3$) etc. have a cubic perovskite type structure (in the paraelectric state) with chemical formula ABO_3 (figure 2 a, b).
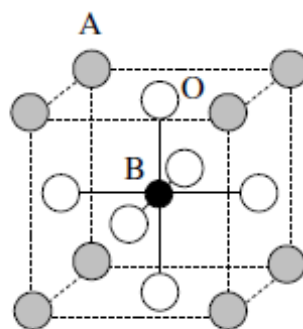

Figure 2 (a).   Perovskite ABO_3 structure with the A and B cations on the corner and body center positions, respectively. Three oxygen anions per unit cell occupy the faces and form octahedra surrounding the B-site.
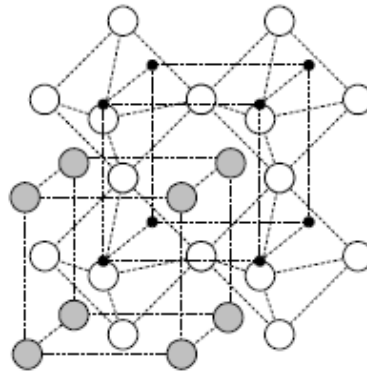
Figure 2 (b)   Perovskite structure (Ba: Grey; Ti: Black; O: White)

As conventionally drawn, A-site cations occupy the corners of a cube, while B-site cations sit at the body center. Three oxygen atoms per unit cell rest on the faces. The lattice constant of these perovskite is always close to the 4 Å due to rigidity of the oxygen octahedral network and the well-defined oxygen ionic radius of 1.35 Å.

A practical advantage of the perovskites structure is that many different cations can be substituted on both the A and B sites without drastically changing the overall structure. Complete solid solutions are easily formed between many cations, often across the entire range of composition. Even though two cations are compatible in solution, their behavior can be radically different when apart from each other. Thus, it is possible to manipulate a material's properties such as Curie Temperature or dielectric constant with only a small substitution of a given cation.

All ferroelectric materials have a transition temperature called the Curie point (T_c). At a temperature $T > T_c$ the crystal does not exhibit ferroelectricity, while for $T < T_c$ it is ferroelectric. On decreasing the temperature through the Curie point, a ferroelectric crystal undergoes a phase transition from a non-ferroelectric (paraelectric) phase to a ferroelectric phase.

Barium Titanate (BaTiO$_3$, BT)

Barium Titanate (BaTiO$_3$) has a ferroelectric tetragonal phase (Fig-3(a)) below its curie point of about 120°C and paraelectric cubic phase (Fig-3(b)) above Curie point. The temperature of the curie point appreciably depends on the impurities present in the sample and the synthesis process.
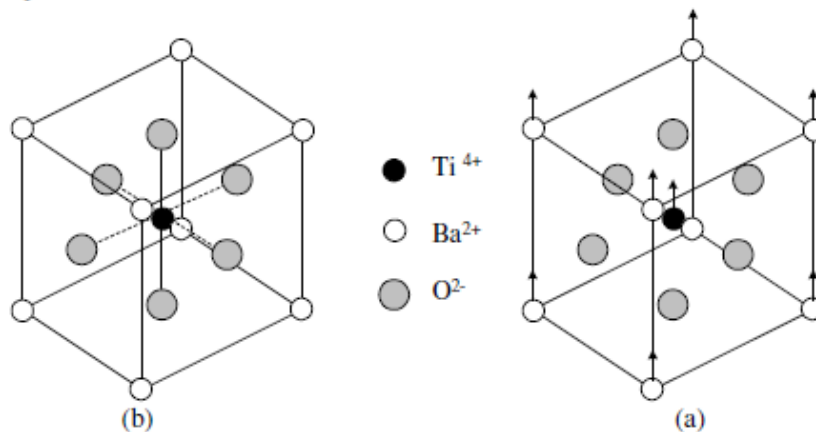

Fig 3 A Perovskite unit cell and the displacements in its ions on the application of an electric field.

In the paraelectric cubic phase the center of positive charges (Ba^{2+}, Ti^{4+}) coincid with the center of negative charges (O^{2-} ion) and on cooling below T_c, a tetragonal phase develops where the center of Ba^{2+} and Ti^{4+} ions are displaced relative to the O^{2-} ions, leading to the formation of electric dipoles.

As the BT ceramics have a very large room temperature dielectric constant, they are mainly used in multilayer capacitor applications. The grain size control is very important for these applications.

Dielectric Constant

The dielectric constant (ε) of a dielectric material can be defined as the ratio of the capacitance using that material as the dielectric in a capacitor to the capacitance using a vacuum as the dielectric. Typical values of ε for dielectrics are:

Material	DIELECTRIC CONSTANT (ε)
Vacuum	1.000
Dry Air	1.0059
Barium Titanate	100-1250
Glass	3.8-14.5
Quartz	5
Mica	4-9
Water distilled	34-78
Soil dry	2.4-2.9
Titanium dioxide	100

Dielectric constant (ε) is given by

$$\varepsilon = \frac{C}{C_0}, \quad C_0 = \frac{\varepsilon_0 A}{t}$$

Where

C = capacitance using the material as the dielectric in the capacitor,
C_0 = capacitance using vacuum as the dielectric
ε_0 = Permittivity of free space (8.85×10^{-12} F/m)
A = Area of the plate/ sample cross section area
t = Thickness of the sample

Brief Description of the Apparatus

1. **Probe Arrangement**

   It has two spring loaded probes. These probe move in pipes and are insulated by teflon bush, which ensure a good electrical insulation. The probe arrangement is mounted in suitable stand, which also hold the sample plate and RTD sensor. The RTD is mounted in the sample plates such that it is just below the sample, separated by a very thin sheet of mica. This ensures the correct measurement of sample temperature. This stand also serves as a lid of the oven. The leads are provided for the connection to RTD and capacitance meter.

STUDY OF DIELECTRIC CONSTANT SETUP

2. Sample

Barium Titanate (BaTiO$_3$) plate with top and bottom conducting surface.

3. Oven

This is a high quality temperature controlled oven. The oven has been designed for fast heating and cooling rates, which enhance the effectiveness of the controller.

4. Main Units

The Set-up consists of two units housed in the same cabinet.

(i) Oven Controller

Platinum RTD (A class) has been used for sensing the temperature. A Wheatstone bridge and an instrumentation amplifier are used for signal conditioning. Feedback circuit ensures offset and linearity trimming and a fast accurate control of the oven temperature.

| Specifications of the Oven	
Temperature Range	: Ambient to 200 ℃
Resolution	: 0.1 ℃
Stability	: ±.1 ℃
Measurement Accuracy	: ± 0.5 ℃
Oven	: Specially designed for Dielectric measurement
Sensor	: RTD (A class)
Display	: 3½ digit, 7 segment LED with autopolarity and decimal indication
Power	: 150W

(ii) Digital Capacitance Meter

This a compact direct reading instrument for the measurement of capacitance of the sample.

Specifications of the Oven	
Range	: 50 to 6000 pf
Resolution	: 1pf
Display	: 3½ digit, 7 segment LED

Experimental Procedure

1. Put a small piece of aluminum foil on the base plate. Pull the spring loaded probes upward, insert the aluminum foil and let them rest on it. Put the sample (BaTiO$_3$) on the foil. Again pull the top of one of the probe and insert the sample below it and let it rest on it gently. Now one of the probes would be in contact with the upper surface of the sample, while the other would be in contact with the lower surface through aluminum foil.

2. Connect the probe leads to the capacitance meter.

3. Connect the oven to the main unit and put the oven in OFF position.

4. Switch on the main unit and note the value of capacitance. It should be a stable reading and is obtained directly in pf.

DIELECTRIC CONSTANT
SAMPLE : Barium Titanate
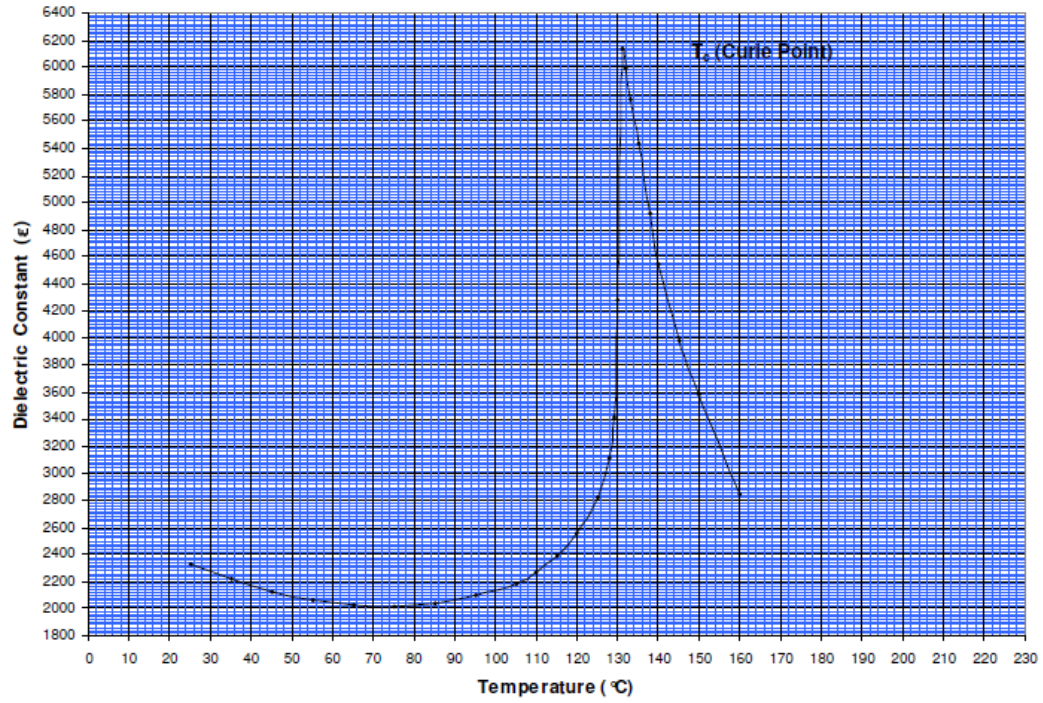
5. (i) Switch ON the temperature Controller and approx adjust the set-temperature. The green LED would light up indicating the oven is ON and temperature would start rising. The temperature of the oven in °C would be indicated by the DPM.

(ii) The controller of the oven would switch ON/OFF power corresponding to set-temperature. In case it is less then the desired, the set-temperature may be increased or vice versa.

(iii) Because of thermal inertia of oven, there would be some over shoot and under shoot before a steady set-temperature is attained and may take 10 minutes for each reading.

(iv) To save time, it is recommended to under adjust the temperature. Example, it is desired to set at 50°C, adjust the temperature set knob so that LED is OFF at 45°C. The temperature would continue to rise. When it reaches 50°C adjust the temperature set knob so that oven is just ON/OFF. It may go up 1 & 2°C, but would settle down to 50°C. Since the change in temperature at this stage is very slow and response of RTD and sample is fast, the reading can also be taken corresponding to any temperature without waiting for a steady state.

Observations and Calculations

Sample : Barium Titanate ($BaTiO_3$)

Area (A) : 8 x 6 mm

Thickness (t) : 1.42 mm

Permittivity of Space (ε_0) : 8.85 x 10^{-12} F/m or 8.85 x 10^{-3} pf/ mm

$$\varepsilon = \frac{C}{C_0} \; ; \; \text{where,} \; C_0 = \frac{\varepsilon_0 A}{t} \Rightarrow \frac{8.85 \times 10^{-3} \times 48}{1.42} \Rightarrow 29.9 \times 10^{-3} \text{ pf}$$

S.No.	Temperature (°C)	Capacitance, C (pf)	Dielectric Constant, (ε)
1	25	696	2328
2	35	665	2224
3	45	636	2127
4	55	618	2067
5	65	607	2030
6	75	604	2020
7	85	612	2047
8	95	629	2104
9	105	654	2187
10	110	677	2264
11	115	715	2391
12	120	764	2555
13	125	846	2829
14	128	930	3110
15	129	1020	3411
16	130	1280	4281
17	131	1836	6140
18	132	1790	5987
19	133	1722	5759
20	135	1627	5441
21	138	1470	4916

22	140	1360	4548
23	145	1190	3980
24	150	1070	3579
25	160	853	2853

Typical Results

From the graph, **Curie Temperature** $(T_c) = 131°C$

Precautions

(1) The spring loaded probe should be allowed to rest on the sample very gently, other wise it may damage the conducting surface of the sample or even break the sample.

(2) The reading of capacitance meter should be taken when the oven is OFF. This would be indicated by the green LED. In ON position there may be some pick ups.

(3) The reading near the Curie Temperature should be taken at closer intervals, say 1°C.

Reference

(1) Introduction to Solid State Physics – C. Kittel, Wiley Eastern Limited (5th Edition).

Solar cell

Pre Lab Questions:

1. A solar cell is a p-n junction diode, explain the following terms:
   a) Doping a semiconductor
   b) Donor and acceptor semiconductors
   c) Effect of doping on energy band scheme of the semiconductor
   d) Majority and minority current carriers
   e) Depletion region
   f) Diffusion current

2. Describe the optical properties in silicon p-n junction diode and explain:
   a) Photo excitation
   b) Photovoltaic effect
   c) Effect of light frequency and light intensity on the number of carriers
   d) Majority and minority carriers in photoconductive devices

3. How does illumination affects the behavior of the p-n junction under different biasing conditions (forward and reverse bias)

4. Describe briefly the components of a solar cell?

5. What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

> ➤ Measure the short-circuit current and no-load voltage at different light intensities and plot the current-voltage characteristic at different light intensities.
> ➤ Determine the Fill factor & the Efficiency of the Solar Cell.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

Characteristic curves of a solar cell

Principle

To measure the current-voltage characteristics of a solar cell at different light intensities, the distance between the light source and the solar cell is varied. Moreover, the dependence of no-load voltage on temperature is determined.

Related topics

Semi-conductor, p-n junction, energy-band diagram, Fermi characteristic energy level, diffusion potential, internal resistance, efficiency, photo-conductive effect, acceptors, donors, valence band, conduction band.

Tasks

1. Measure the short-circuit current and no-load voltage at different light intensities and plot the current-voltage characteristic at different light intensities.

2. Estimate the dependence of no-load voltage and short-circuit current on temperature.

3. Plot the current-voltage characteristic under different operating conditions: cooling the equipment with a blower, no cooling, shining the light through a glass plate.

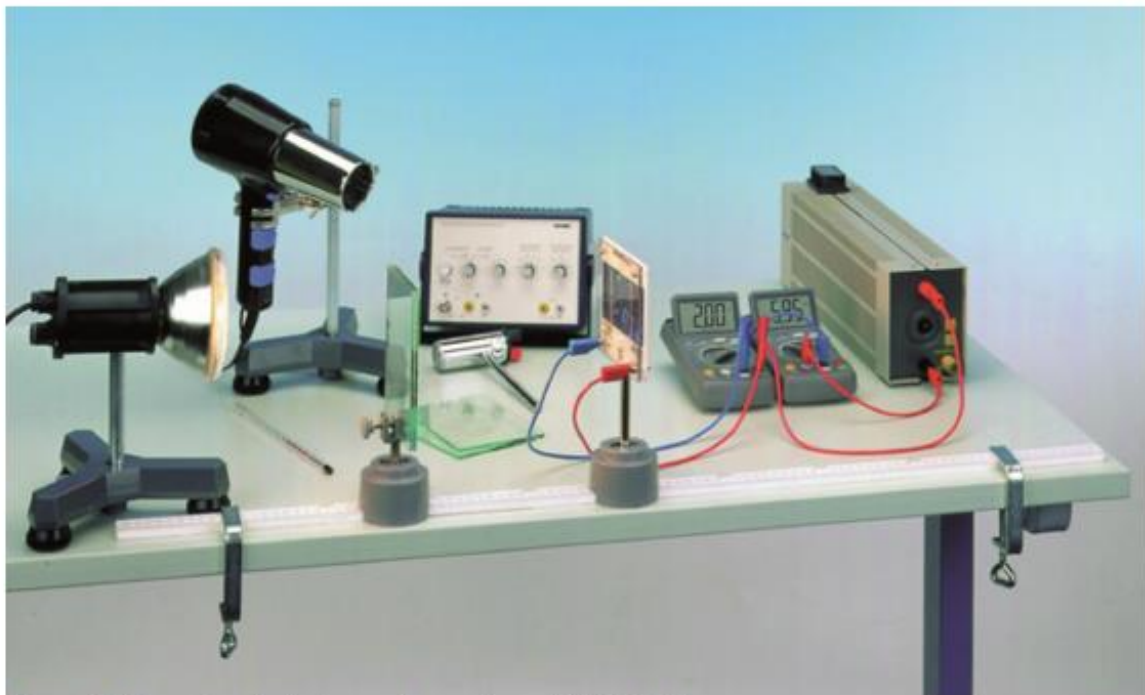4. Determine the characteristic curve when illuminating by sunlight.

Figure 1: Experimental set-up of experiment P2410901.

Equipment

1	Solar battery, 4 cells, 2.55 cm	06752-04
1	Thermopile, molltype	08479-00
1	Universal measuring amplifier	13626-93
1	Rheostat, 330 Ohm, 1.0 A	06116-02
1	Lamp socket E27, mains conn.	06751-00
1	Filament lamp, 220 V/120 W, w. refl.	06759-93
1	Hot-/Cold air blower, 1700 W	04030-93
1	Meter scale, demo, l = 1000 mm	03001-00
2	Tripod base -PASS-	02002-55
2	Barrel base -PASS-	02006-55
2	Support rod -PASS-, square, l = 250 mm	02025-55
2	Right angle clamp -PASS-	02040-55
1	Plate holder	02062-00
1	Universal clamp	37715-00
2	Bench clamp -PASS-	02010-00
1	Glass pane, 1501004 mm, 2 off	35010-10
2	Digital multimeter	07134-00
1	Lab thermometer, -10...+100°C	38056-00
3	Connecting cord, l = 500 mm, red	07361-01
2	Connecting cord, l = 500 mm, blue	07361-04

Set-up and procedure

- The thermopile only measures the light of the lamp but the solar cell also detects the diffused light coming from reflections on the bench top. Therefore, it is recommended to cover the bench with a black cloth or piece of black card to suppress the diffused light.

- The experimental set-up is as shown in Fig. 1. The glass plate is only needed for task 3.

- Do the electrical connections as in Fig. 2

Figure 2: Circuit for measuring the current-voltage characteristic.

Task1

The light intensity is varied by varying the distance between the light source and the solar cell. First of all, measure the light intensity with the thermopile and amplifier with the equipment at different distances from the light source. (**Note**: the maximum output voltage of the amplifier is 10 V). The inlet aperture marks the position of the thermopile. The distance between the lamp and the thermopile should be at least 50 cm, since the angular aperture of the thermopile is only 20°.

To suppress the influence of the temperature on the characteristics of the solar cell, keep it at room temperature with the aid of the cold air blower during the experiment.
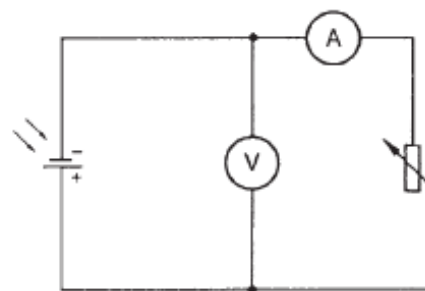
Task 2

To demonstrate the temperature effect, blow hot air over the solar cell and measure the temperature directly in front of it with a thermometer. Do not touch the cell as its thin p-layer can easily be damaged.

We recommend separating the lamp and solar cell more than 50 cm, because in shorter distances the temperature rise caused by radiation could falsify the measurement. Measure the noload voltage and the short-circuit current.

Task 4

- The characteristics of the solar cell should be measured in sunlight also if possible; in this case both direct and diffused light are involved.

- The thermophile is used again to determine the relationship between the short-circuit current and the light intensity, although it measures only direct light because of its small angular aperture. For comparative purposes, therefore, we must support a black cardboard tube about 20 cm long in front of the solar cell to screen it from the diffused light. It is important that the thermopile and the solar cell are pointing directly into the sun.

Theory and evaluation

Pure silicon is deliberately 'impurified' (doped) with tri- and pentavalent impurity atoms to make a p- or n-type semi-conductor. If we put a p-and n-type crystal together we get a junction (pn-junction, Fig. 3) whose electrical properties determine the performance of the solar cell.

In equilibrium (with no external voltage) the Fermi characteristic energy level E_F will be the same throughout. Because of the difference in the concentrations of electrons and holes in the p- and n-regions, electrons diffuse into the p-region and holes into the n-region. The immobile impurity atoms create a space charge-limited current region; the diffusion current and the field current offset one another in equilibrium.

The diffusion potential U_D in the pn-junction depends on the amount of doping and corresponds to the original difference between the Fermi energy levels of the separate p- and n-regions.

The distance between the valence band and the conduction band in silicon at room temperature is

$$E = 1.1 \text{ eV}$$

For silicon, the diffusion potential is

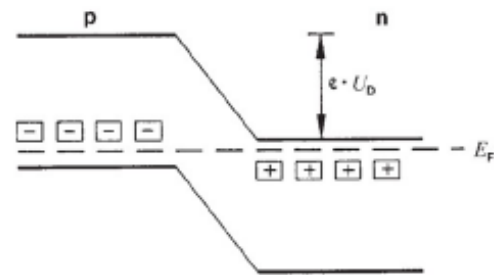$$U_D = 0.5 \text{ to } 0.7 \text{ V}.$$

Figure 3: : pn-junction in the energy-band diagram – acceptors, + donors, U_D is the diffusion potential, E_F is the Fermi characteristic energy level, and e is the elementary charge.
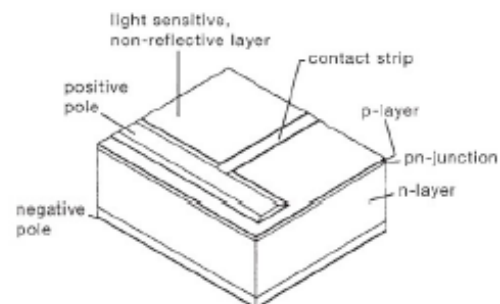

Figure 4: Construction of a silicon solar cell.

If light falls on the pn-junction, the photons create electronhole pairs separated by the space charge. The electrons are drawn into the n-region and the holes into the p-region. Photons are absorbed not only in the pn-junction but also in the p-layer above it. The electrons produced are minority carriers in those areas: their concentration is greatly reduced by recombination and with it their efficiency. The p-layer must therefore be sufficiently thin for the electrons of diffusion length L_E to enter the n-layer.

$$L_E \gg t$$

where t = thickness of p-layer.

If g is the number of electron-hole pairs produced per unit area and of a voltage U is applied across the pn-junction, a stream of electrons and holes of density

$$i = e \cdot \left(e^{eU/kT-1}\right) \cdot \left(\frac{n_0 D_e t}{L^2 e} + \frac{p_0 D_h}{L_h}\right) - eg \tag{1}$$

is produced, where e is the elementary charge, k is Boltzmann's constant, T is the temperature, L is the diffusion length of electrons and holes, D is the diffusion constant for electrons and holes, n_0 and p_0 are equilibrium concentrations of the minority carriers. The short-circuit current density ($U = 0$)

$$i_s = -e \cdot g \tag{2}$$

is proportional to the intensity of the incident light at fixed temperature. g becomes very slightly greater (less than 0.01 %/K) as the temperature rises.

The voltage U can become as high as the diffusion potential U_D but no higher. As the temperature rises the no-load voltage decreases typically by –2.3 mV/K, since the equilibrium concentrations n_0 and p_0 increase with the temperature:

$$n_0 \sim e^{-\frac{\Delta E}{2kT}}$$

Figure 5: Light intensity J at distances s normal to the light source.

Task 1:

For this task, it is assumed that all the light entering the aperture (dia. 2.5 cm) reaches the measuring surface.

The sensitivity is 0.16 mv/mW. Plotting the light intensity J over the distance s gives a straight line. By extrapolating the straight line we can determine the intensity at distances $s \leq 50$ cm.

Fig 6 shows the relationship between the light intensity and the short-circuit current and no-load voltage (Fig. 6).

The solar battery which consists of four cells connected in series thus has a maximum no-load voltage of 2 V. The shortcircuit current is proportional to the light intensity.
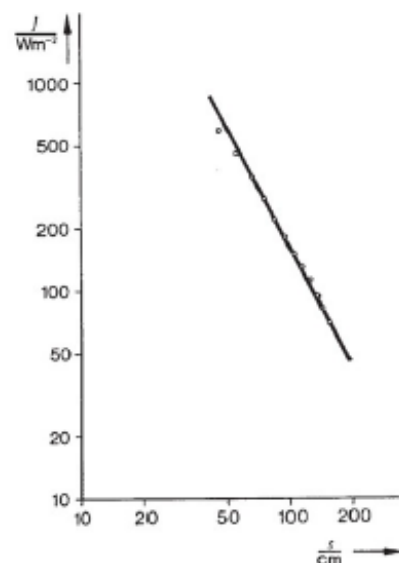
$$I_s = 1.84 \cdot 10^{-4} \, \text{A/Wm}^{-2} \cdot J$$

The current-voltage characteristic at different light intensities J is shown in Fig. 7. The maximum power output is at the turning points on the curves (joined by the broken line; Fig. 7) at which the load resistor has the same value as the internal resistance R_i of the solar battery.

The internal resistance decreases with increasing light intensity. If we compare the maximum power output with the incident power, we obtain an efficiency of approx. 6% (area of solar battery 50 cm^2).
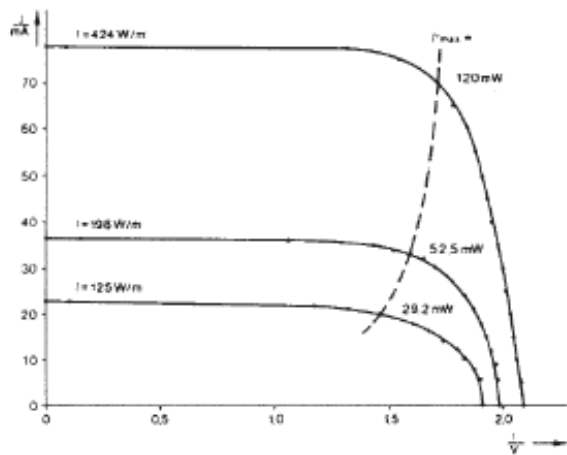

Figure 6: Current-voltage characteristic at different light intensities J.

Figure 7: Short-circuit current I_s and no-load voltage U_0 as a function of the light intensity J.

Task 2:

Measuring the effect of temperature on U_0 and I_s the temperature distribution over the hot air area must be taken into account. The measurements can provide only a rough order of magnitude of this. Measuring the no-load voltage with hot and cold air gave:

$$\frac{\Delta U_0}{\Delta T} = -8\,\text{mV/K}$$

We thus obtain the value –2 mV/K for one cell. The change in short-circuit current with the temperature cannot be measured.

Task 3:

A glass plate which absorbs light in the infrared region can be used to reduce a rise in temperature of the solar battery. Fig. 8 shows the effect of the various "operating modes'"
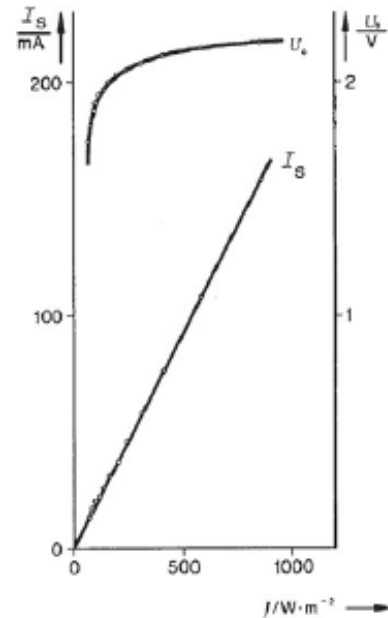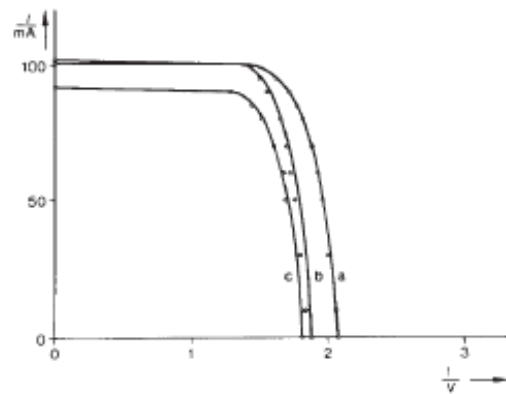

Fig. 8: Current-voltage characteristics of the solar battery a) with blower cooling b) with no blower cooling c) when screened with a glass plate.

Task 4:

Sunlight incident on solar cells produces different characteristic curves from incandescent light. The reason lies in the different spectra of the two light sources (Fig. 9). At the same light intensity, sunlight produces a higher shortcircuit current

$$I_s = 3.04 \cdot 10^{-4} \, \text{JA/Wm}^{-2}$$

Because the infrared region of the spectrum of sunlight is smaller, the solar cell does not heat up so much and the measurements with and without cooling provide the same characteristics for sunlight.
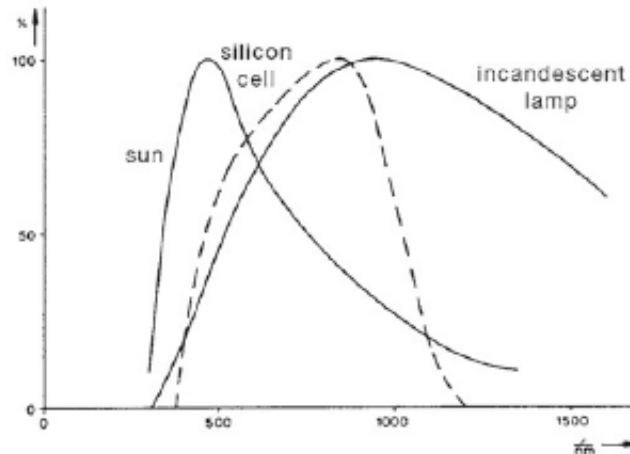

Fig. 9: Spectrum of the sun (T approx. 5800 K) and of an incandescent lamp (T approx. 2000 K), and the spectral sensitivity of the silicon solar cell.

Seebeck effect

Pre Lab Questions:

1- What is thermoelectric effect?
2- What is the relation between Seebeck effect, Peltier effect, and Thomson effect?
3- Why do different metals have different Seebeck coefficients?
4- What is your goal for the experiment and how are you going to achieve it?

Seebeck effect

The aims of this experiment are:

➢ Obtain the electrical power P_{el}.

➢ Obtain the thermal power and measure the efficiency of the thermoelectric generator.

➢ Obtain the Seebeck Coefficient.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

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Semiconductor thermogenerator

Related topics

Seebeck effect (thermoelectric effect), thermoelectric e.m.f., efficiency, Peltier coefficient, Thomson coefficient, Seebeck coefficient, direct energy conversion, Thomson equations.

Principle

In a semi-conductor thermogenerator, the no-load voltage and the short-circuit current are measured as a function of the temperature difference. The internal resistance, the Seebeck coefficient and the efficiency are determined.

Equipment

Thermogenerator	04366.00	1
Flow-through heat exchanger	04366.01	2
Heat conductive paste, 50 g	03747.00	1
Connection box	06030.23	1
Rheostat, 33 Ohm, 3.1 A	06112.02	1
Voltmeter, 0.3-300 VDC, 10-300 VAC	07035.00	1
Ammeter 1/5 A DC	07038.00	1
Stopwatch, digital, 1/100 sec.	03071.01	1
Immersion thermostat TC10	08492.93	1
Accessory set for TC10	08492.01	1
Bath for thermostat, Makrolon	08487.02	1
Lab thermometer, -10...+100°C	38056.00	1
Thermometer, -10...+ 50°C	38033.00	1
Resistor 3.3 Ohm	39104.25	1
Rubber tubing, i.d. 6 mm	39282.00	4
Connecting cord, l = 500 mm, red	07361.01	3
Connecting cord, l = 500 mm, blue	07361.04	2

Tasks

1. To measure no-load voltage U_0 and short-circuit current I_s at different temperature differences and to determine the Seebeck coefficient.

2. To measure current and voltage at a constant temperature difference but with different load resistors, and to determine the internal resistance R_i from the measured values.

3. To determine the efficiency of energy conversion, from the quantity of heat consumed and the electrical energy produced per unit time.

Set-up and procedure

The experiment is set up as shown in Fig. 1.

1. Secure flow-type heat exchangers to each side of the thermogenerator. Fill the cold side with tap water and set the temperature of the hot side on the thermostat. The two temperatures are measured using the holes in the thermogenerator provided for the purpose. The short-circuit current and the no-load voltage are measured directly, the internal resistance of the measuring equipment being disregarded.

Fig. 1: Experimental set-up for measuring no-load voltage and short-circuit current as a function of temperature difference.

Fig. 2: Construction of a semiconductor Seebeck element. Several elements are generally connected electrically in series and thermally in parallel.
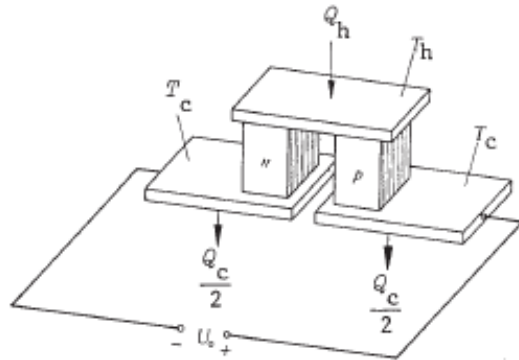
2. Connect rheostat R_{ext} to the thermogenerator at a constant average temperature difference. Measure the current and voltage at different settings and plot the results on a graph.

3. Remove the heat exchanger which was connected to the thermostat and put a water bath brim-full of boiling water in its place. Measure the temperature of the hot side $T_h = f(t)$ and of the cold side $T_c = f(t)$ as a function of time. Measure the current and the voltage across an external resistance of approximately the same value as the internal resistance.

Theory and evaluation

If a temperature drop is created along a current-free branch of a conductor made up of different materials, heat flows from the warmer region to the cooler one. The charge carriers which take part in this transfer of heat are unevenly distributed along the conductor. An internal field strength is set up, which can be shown to be the e.m.f. U_0 at the open ends of the conductor (Seebeck effect).

The voltage level depends on the temperature difference and on the materials used. To a first approximation, the voltage may be written:

$$U_0 = \alpha_{1,2} (T_h - T_c) = \alpha_{1,2} \Delta T$$

where $\alpha_{1,2}$ is the Seebeck coefficient of the combination of materials used, T_h is the temperature of the hot side and T_c the temperature of the cold side.

1. Applying the regression expression
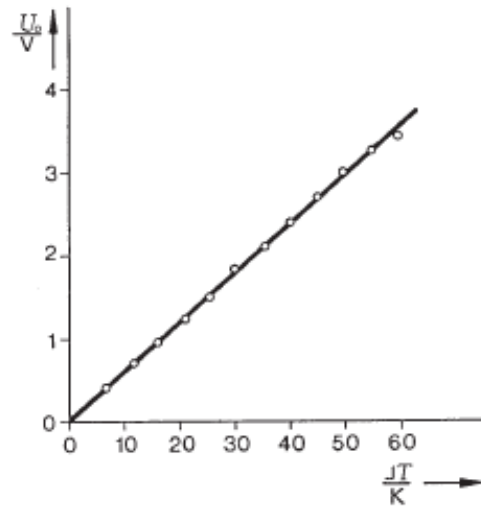
$$U_0 = a + b \Delta T$$

to the measured values in Fig. 3, we obtain

$$b = 0.0587 \frac{V}{K}$$

with the standard error

$$s_b = 0.0006 \frac{V}{K}$$

Fig. 3: No-load voltage as a function of the temperature difference.

The thermogenerator consists of 142 elements connected in series. The Seebeck coefficient of the semiconductor combination used is therefore

$$\alpha_{1,2} = 4.13 \cdot 10^{-4} \frac{V}{K}$$

with the standard error

$$s_{\alpha_{1,2}} = 4.04 \cdot 10^{-4} \frac{V}{K}$$

As the short-circuit also increases linearly with the temperature, the internal resistance of the thermogenerator is constant in the temperature range considered.
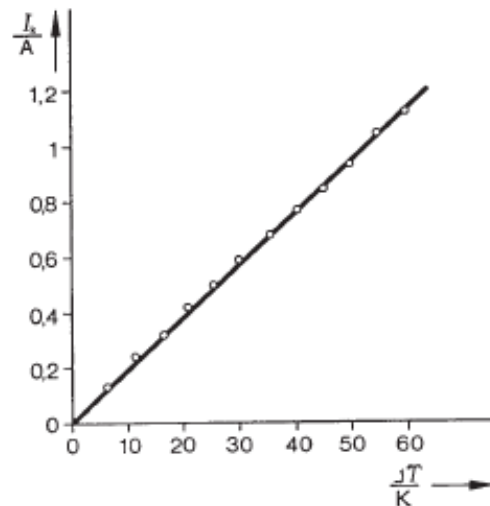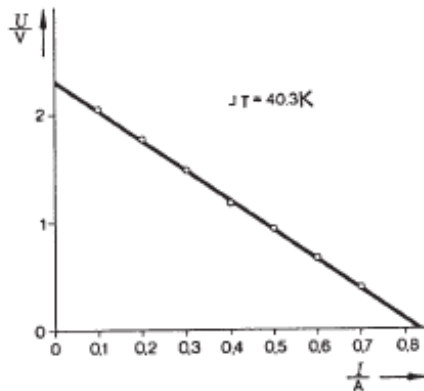

Fig. 4: Short-circuit current as a function of the temperature difference.

Fig. 5: Terminal voltage as a function of the current strength of a constant temperature difference.

2. Applying the regression expression $U = a + b\,I$ to the measured values from Fig. 5 we obtain

$$a = U_0 = 2.34\ \text{V}$$
$$s_a = s_{U_0} = 0.01\ \text{V}$$
$$\text{and}\quad |b| = R_I = 2.80\ \Omega$$
$$s_b = s_{R_I} = 0.02\ \Omega.$$

and the short circuit current

$$I_S = \frac{U_0}{R_I} = 0.84\ \text{A}$$

with $s_{I_S} = 0.01\ \text{A}$

3. From Fig. 6 we determine the slope of the (descending) curve at one point by drawing a tangent or by linear regression.
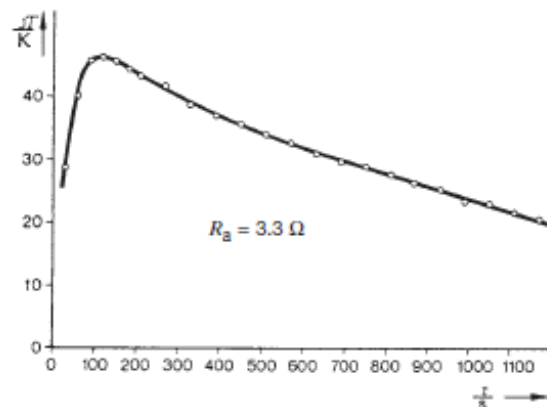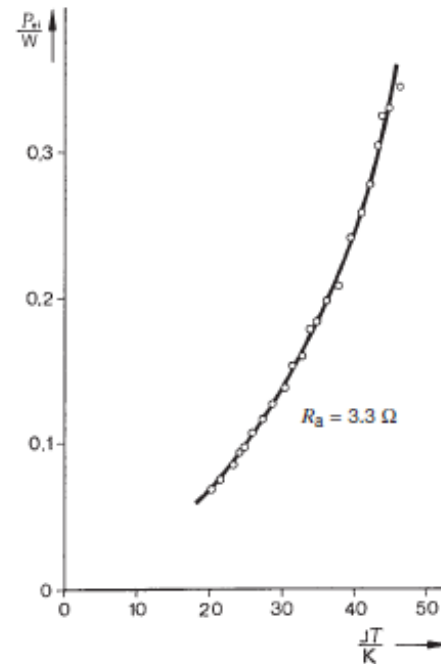

Fig. 6: Temperature difference as a function of time.

Fig. 7: Electrical power generated as a function of the temperature difference.

At a temperature difference ΔT of 40 K we obtain the following for the nearest measured values, using the regression expression $\Delta T = a + b\,t$:

$$b = \frac{\text{d}\Delta T}{\text{d}t} = -0.0361\ \frac{\text{K}}{\text{s}}$$

We can thus work out the quantity of heat Q flowing through the generator in unit time in accordance with

$$\frac{\text{d}Q}{\text{d}t} = P_{\text{th.}} = C \cdot \frac{\text{d}\Delta T}{\text{d}t}$$

As the mass of water $m_W = 0.194$ kg and the specific heat capacity if water

$$c_W = 4182\ \frac{\text{J}}{\text{K}}$$

we obtain

$$C = m_W \cdot c_W = 811\ \frac{\text{J}}{\text{Kg K}}$$

so that

$$P_{\text{th.}} = 29.3\ \frac{\text{J}}{\text{s}}$$

The electrical power, measured at constant load, $P_{\text{el.}}$, can be obtained from Fig. 7. For a temperature difference $\Delta T = 40$ K we obtain $P_{\text{el.}} = 0.25$ W, so that the efficiency

$$\eta = \frac{P_{\text{el.}}}{P_{\text{th.}}} = = 0.009\ \text{or}\ 0.9\,\%$$

X-ray Diffraction

Pre Lab Questions:

1- Explain Bragg's law and drive its equation?
2- Explain how x-rays are produced, and what voltage and current ranges are involved in laboratory x-ray unit?
3- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➢ Investigating Bragg reflection at a NaCl monocrystal.

➢ Determining the lattice constant a_0 of NaCl.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

Properties of crystals & X-ray structural analysis

Bragg reflection:
determining
the lattice constants
of monocrystals

Objects of the experiment

■ Investigating and comparing Bragg reflection at an LiF and an NaCl monocrystal.

■ Determining the lattice constant a_0 of NaCl and LiF.

Principles

Bragg's law of reflection describes the diffraction of plane waves at a monocrystal as the selective reflection of the waves at a set of lattice planes within the crystal. Due to the periodicity of the crystal, the lattice planes of a set have a fixed spacing d. An incident wave with the wavelength λ is reflected with maximum intensity when the Bragg condition

$$n \cdot \lambda = 2 \cdot d \cdot \sin\vartheta \qquad (I)$$

n: diffraction order
λ: wavelength
d: spacing of lattice planes

is fulfilled (see experiment P6.3.3.1). The angle ϑ shows the direction of the incident and reflected wave with respect to the

set of lattice planes and is often referred to as the glancing angle.

In a cubic crystal with NaCl structure (cf. Fig. 1), the lattice planes run parallel to the surfaces of the crystal's unit cells in the simplest case. Their spacing d corresponds to one half the lattice constant:

$$d = \frac{a_0}{2} \qquad (II)$$

This lets us use (I) as an equation for determining the lattice constant a_0:

$$n \cdot \lambda = a_0 \cdot \sin\vartheta \qquad (III)$$

In other words, to determine a_0 we need to measure the glancing angle ϑ for a known wavelength λ and diffraction order n. This method is more precise when the glancing angles are also measured in higher diffraction orders.

In this experiment, the molybdenum x-rays are used as radiation of a known wavelength. Table 1 shows its wavelengths λ.

Table 1: Wavelengths of the characteristic x-ray radiation of molybdenum (weighted means [1])

Line	$\dfrac{\lambda}{pm}$
K_α	71.08
K_β	63.09

Fig. 1   Three-dimensional representation of the structure of NaCl
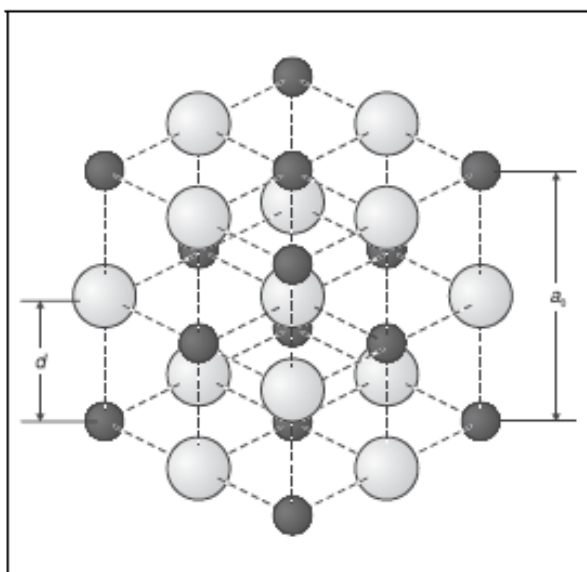d: Spacing of lattice planes in [1,0,0]-direction
a_0: lattice constant

A Geiger-Müller counter tube is used to detect the x-rays; this instrument and the crystal are both pivoted with respect to the incident x-ray beam in 2ϑ coupling – the counter tube is turned by twice the angle of the crystal (cf. Fig. 2). The zero point $\vartheta = 0°$ is characterized by the fact that the lattice planes and the

Apparatus

1 X-ray apparatus	554 811
1 End-window counter for α, β, γ and x-ray radiation	559 01
1 LiF monocrystal for Bragg reflection . . .	554 77

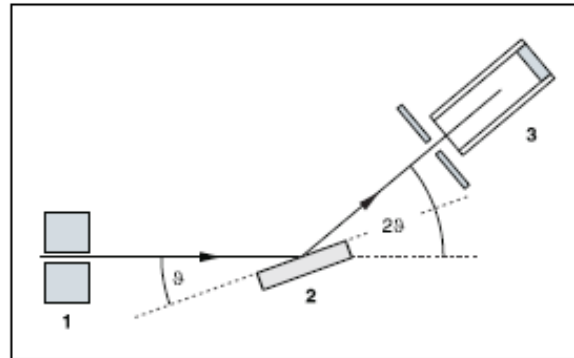*additionally required:*

1 PC with Windows 9x/NT

Fig. 2 Schematic diagram of diffraction of x-rays at a monocrystal and 2ϑ coupling between counter-tube angle and scattering angle (glancing angle)
1 collimator, **2** monocrystal, **3** counter tube

axis of the counter tube are parallel to the incident x-ray beam. As the lattice planes are seldom precisely parallel to the surface of the crystal, the zero point of each crystal must be calibrated individually.

Safety notes

The x-ray apparatus fulfills all regulations governing an x-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807/97 R0).

The built-in protection and screening measures reduce the local dose rate outside of the x-ray apparatus to less than 1 μSv/h, a value which is on the order of magnitude of the natural background radiation.

■ Before putting the x-ray apparatus into operation inspect it for damage and to make sure that the high voltage is shut off when the sliding doors are opened (see Instruction Sheet for x-ray apparatus).

■ Keep the x-ray apparatus secure from access by unauthorized persons.
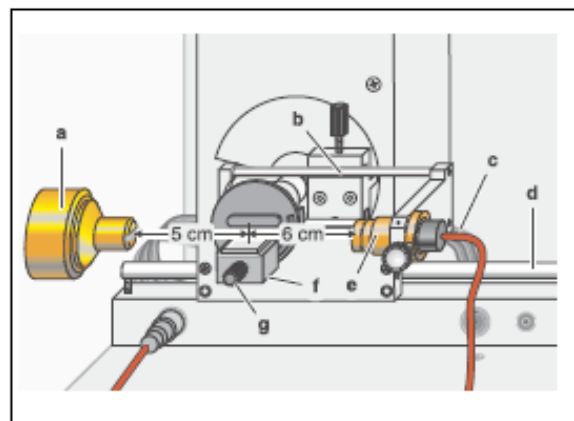
Do not allow the anode of the x-ray tube Mo to overheat.

■ When switching on the x-ray apparatus, check to make sure that the ventilator in the tube chamber is turning.

The goniometer is positioned solely by electric stepper motors.

■ Do not block the target arm and sensor arm of the goniometer and do not use force to move them.

Setup

Setup in Bragg configuration:

Fig. 3 shows some important details of the experiment setup. To set up the experiment, proceed as follows (see also the Instruction Sheet for the x-ray apparatus):

– Mount the collimator in the collimator mount **(a)** (note the guide groove).

– Attach the goniometer to guide rods **(d)** so that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm. Connect ribbon cable **(c)** for controlling the goniometer.

– Remove the protective cap of the end-window counter, place the end-window counter in sensor seat **(e)** and connect the counter tube cable to the socket marked GM TUBE.

Fig. 3 Experiment setup in Bragg configuration

- By moving the sensor holder **(b)**, set the distance s_2 between the target arm and the slit diaphragm of the sensor seat to approx. 6 cm.
- Mount the target holder **(f)** with target stage.
- Manually align the target and sensor arm horizontally using the ADJUST knob and save these positions as the "zero position of the measuring system" by pressing TARGET, COUPLED and β Limits at the same time (see Instruction Sheet for x-ray apparatus).

Preparing the PC-based measurement:

- Connect the RS–232 output and the serial interface on your PC (usually COM1 or COM2) using the 9-pin V.24 cable (supplied with x-ray apparatus).
- If necessary, install the software "X-ray Apparatus" under Windows 9x/NT (see Instruction Sheet for x-ray apparatus) and select the desired language.

Carrying out the experiment

Notes:

NaCl and LiF crystals are hygroscopic and extremely fragile. Store the crystals in a dry place; avoid mechanical stresses on the crystals; handle the crystals by the short faces only.

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor somewhat. However, the distance should not be too small, as otherwise the angular resolution of the goniometer is no longer sufficient to separate the characteristic K_α and K_β lines.

a) Bragg reflection at an LiF monocrystal:

- Loosen knurled screw **(g)**, place the LiF crystal flat on the target stage, carefully raise the target stage with crystal all the way to the stop and gently tighten the knurled screw (prevent skewing of the crystal by applying a slight pressure).
- Set the tube high voltage U = 35.0 kV and the emission current I = 1.00 mA.

Determining the zero position of the measuring system

- In coupled scanning mode, set the target to about 10.2° using the ADJUST knob.
- Switch on the tube high voltage with HV on/off.
- Leave the target position unchanged and, in sensor scanning mode, manually find the counting rate maximum for the first reflection maximum of the K_α line.
- Leave the sensor unchanged in the maximum counting-rate position and manually find the maximum of the counting rate in target mode.
- Switch between sensor and target modes and check whether you have found the counting rate maximum.

- In coupled scanning mode, move the target back by 10.2° (even if this takes you into the negative range!).
- Save the positions of the target and the sensor as the "zero position of the measuring system" by pressing TARGET, COUPLED and β LIMITS simultaneously.

Recording the diffraction spectrum:

- Start the software "X-ray Apparatus", check to make sure that the apparatus is connected correctly, and clear any existing measurement data using the button ☐ or the F4 key.
- Set the measuring time per angular step Δt = 10 s and the angular step width $\Delta \beta$ = 0.1°.
- Press the COUPLED key to activate 2ϑ coupling of target and sensor and set the lower limit of the target angle to 4° and the upper limit to 34°.
- Start measurement and data transfer to the PC by pressing the SCAN key.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the button ☐ or the F2 key.

a) Bragg reflection at an NaCl monocrystal:

- Press the ZERO key to return the target and sensor to the current zero position.
- Remove the LiF crystal and carefully mount the NaCl crystal in its place.

Determining the zero position of the measuring system

- In coupled scanning mode, set the target to about 7.2° using the ADJUST knob.
- Switch on the tube high voltage with HV on/off.
- Leave the target position unchanged and, in sensor scanning mode, manually find the counting rate maximum for the first reflection maximum of the K_α line.
- Leave the sensor unchanged in the maximum counting-rate position and manually find the maximum of the counting rate in target mode.
- Switch between sensor and target modes and check whether you have found the counting rate maximum.
- In coupled scanning mode, move the target back by 7.2° (even if this takes you into the negative range!).
- Save the positions of the target and the sensor as the "zero position of the measuring system" by pressing TARGET, COUPLED and β LIMITS simultaneously.

Recording the diffraction spectrum:

- Start the software "X-ray Apparatus" or clear any existing measurement data using the button or the ☐ F4 key.
- Press the COUPLED key to activate 2ϑ coupling of target and sensor and set the lower limit of the target angle to 4° and the upper limit to 24°.
- Start measurement and data transfer to the PC by pressing the SCAN key.
- When you have finished measuring, save the measurement series under an appropriate name by pressing the button ☐ or the F2 key.

Measuring example

a) Bragg reflection at an LiF monocrystal:

Fig. 4 Diffraction spectrum of x-rays in Bragg reflection to the third diffraction order at an LiF monocrystal with logarithmic display of counting rate R.
Parameters of x-ray tube:
$U = 35$ kV, $I = 1$ mA

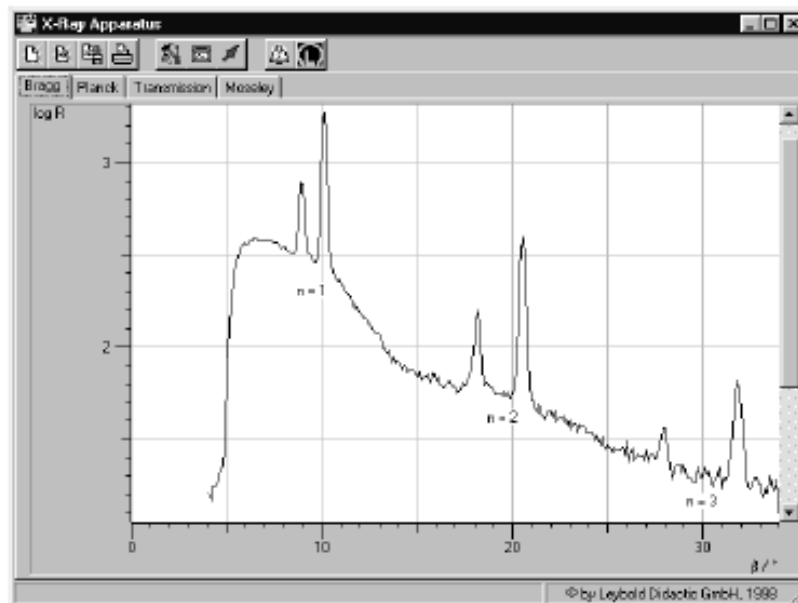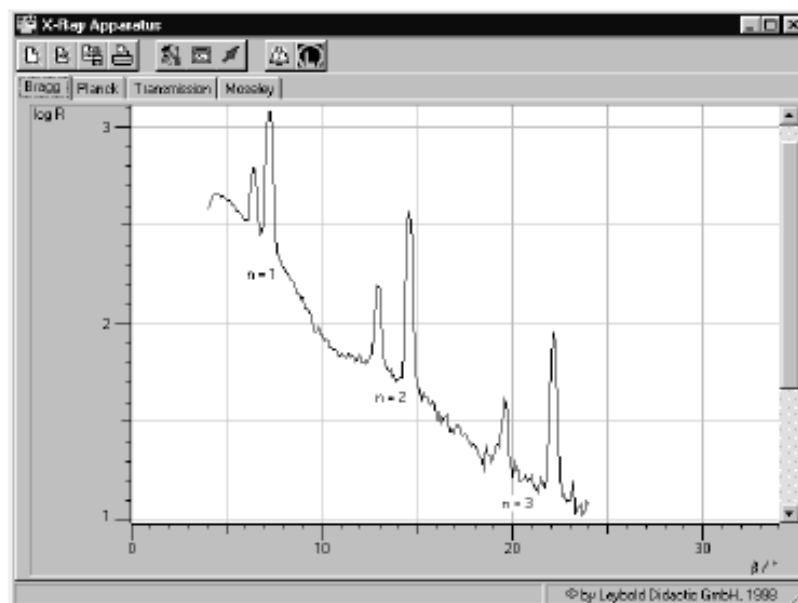### b) Bragg reflection at an NaCl monocrystal:

Fig. 5 Diffraction spectrum of x-rays in Bragg reflection to the third diffraction order at an NaCl monocrystal with logarithmic display of counting rate R.
Parameters of x-ray tube:
$U = 35$ kV, $I = 1$ mA

Evaluation

– In each diagram, click the right mouse button to access the evaluation functions of the software "X-ray Apparatus" and select the command "Calculate Peak Center" to evaluate the diffraction spectra.

– Using the left mouse button, mark the "full width" of each peak and write down the center values in a table as the glancing angle (see tables 2 and 3).

– For each glancing angle ϑ, calculate the values $\sin \vartheta$ and and plot these value pairs in a diagram (see Fig. 6).

In each case, the results lie along a straight line through the origin; in accordance with (III), its slope corresponds to the lattice constant a_0.
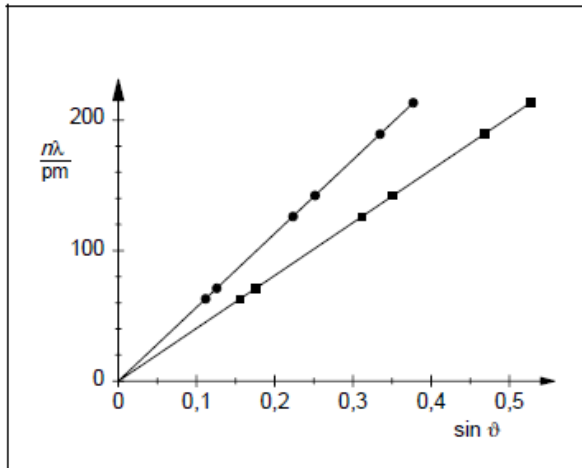
Fig. 6   Value pairs as a function of sin ϑ
LiF: squares, slope of line = 404.5 pm
NaCl: circles, slope of line = 565.2 pm

Table 3: Glancing angle ϑ of the NaCl crystal

ϑ	sin ϑ	Line	n	$\dfrac{n \cdot \lambda}{pm}$
6.41°	0.112	K_β	1	63.06
7.23°	0.126	K_α	1	71.08
12.91°	0.223	K_β	2	126.12
14.57°	0.252	K_α	2	142.16
19.55°	0.335	K_β	3	189.18
22.15°	0.377	K_α	3	213.24

Table 2: Glancing angle ϑ of the LiF crystal

ϑ	sin ϑ	Line	n	$\dfrac{n \cdot \lambda}{pm}$
8.95°	0.156	K_β	1	63.06
10.10°	0.175	K_α	1	71.08
18.17°	0.312	K_β	2	126.12
20.54°	0.351	K_α	2	142.16
27.91°	0.468	K_β	3	189.18
31.82°	0.527	K_α	3	213.24

Results

a) LiF crystal:

Measurement result:	a_0 = 404.5 pm
Literature value [2]:	a_0 = 402.7 pm
Ion radii [3]:	68 pm (Li$^+$), 133 pm (F$^-$)
Sum of ion radii:	201 pm

b) NaCl crystal:

Measurement result:	a_0 = 565.2 pm
Literature value:	a_0 = 564.02 pm
Ion radii [3]:	98 pm (Na$^+$), 181 pm (Cl$^-$)
Sum of ion radii:	279 pm

Conclusion: the LiF lattice shows a significantly smaller lattice constant than the NaCl lattice, as the radii of the ions involved are smaller.

Literature

[1] C. M. Lederer and V. S. Shirley, Table of Isotopes, 7th Edition, 1978, John Wiley & Sons, Inc., New York, USA.

[2] Handbook of Chemistry and Physics, 52nd Edition (1971–72), The Chemical Rubber Company, Cleveland, Ohio, USA.

[3] Charles Kittel, Introduction to Solid State Physics, John Wiley & Sons, Inc. New York, USA

X-ray fluorescence (XRF)

Pre Lab Questions:

1- How does x-ray fluorescence happen?

2- What does K,L,M and α, β, mean in the names of the spectral lines in XRF?

3- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➤ Determination of the elements present in an alloy.

➤ Determination of the chemical composition and concentrations of a brass sample.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy t the experiment sheets!

Finally:

Discus your results!

X-ray fluorescence analysis

Determination of the chemical composition of a brass sample by x-ray fluorescence analysis

Objects of the experiment
- Recording the x-ray fluorescence spectrum of a brass sample.
- Determining the mass ratios of individual alloy components.

Principles

A sample irradiated with high-energy x-ray photons emits characteristic x-ray lines, whose energy depends on the atomic number of the element of the sample material. This dependence (Moseley's law) together with the x-ray fluorescence spectra of several elements was examined in the LD Physics Leaflets P6.3.5.4 and P6.3.5.5.

If the sample represents a chemical compound or mixture then its fluorescence spectrum is also of a complex nature. Since the inner electron shells (between which the x-ray transitions occur) are not incorporated in the chemical bond, the characteristic lines are also to a large degree *independent of the chemical bond of the element*. Hence the x-ray fluorescence spectra of a chemical compound are, as a first approximation, a superposition of spectra of its components.

To make a qualitative analysis of the chemical composition of a sample, all of the existing peaks in the fluorescence spectrum are firstly assigned to the elements. This is performed with the help of tabular values for the energies of the characteristic lines. In making the assignment the "pattern" of each spectral series is also considered: thus as well as the $K\alpha$ line, the $K\beta$ line with smaller (ca. 5 to 10 times) intensity must also be in the spectrum; the $L\alpha$ line appears together with the $L\beta$ line with similar intensity and the $L\gamma$ line with smaller intensity.

Conclusions can be made about the relative proportions of each element in the compound on the basis of the relative intensities of their fluorescent lines.

To make a quantitative analysis however, many other factors need to be taken into account, factors which influence the relationship of the line intensities of various elements. Matrix effects for example play a large role, whereby "matrix" here is understood to mean the physical and chemical neighbourhood of the element under scrutiny. Also the surface quality and the geometric properties of the sample need to be considered. Furthermore the influence of the measuring system as well as the detector response probability and the dead time all need to be accounted for.

Since the intensity of the peaks depends not only on the concentration of the element but also on the position of its absorption discontinuities relative to the excitation spectrum, the matrix effects also occur via the absorption (especially the discontinuity absorption) of the primary radiation through the other elements of the compound. The fluorescence radiation of the element under scrutiny is also in part absorbed by the matrix to a considerable degree, before reaching the detector.

In this experiment, the quantitative analysis of the chemical composition of a brass sample containing lead is carried out. The components in this alloy were already identified in the LD Physics Leaflet P7.5.1.1. The matrix effects are not considered.

For calculating the mass ratios, the fact is made use of that the height of a peak is proportional to the number n of radiating atoms. In the reference spectrum this number n_0 is determined by the density of the substance ρ, its atomic weight A, the radiated area S and the effective thickness d of the irradiated layer:

$$n_0 = S \cdot d \cdot \frac{\rho}{A}.$$

For the number of atoms of each type in the alloy, to first approximation the expression

$$n = n_0 \cdot \frac{H}{H_0} = V \cdot \frac{\rho}{A} \cdot \frac{H}{H_0}$$

can be used. In this H and H_0 are the peak heights in the spectrum to be analysed and in the reference spectrum is $V = S \cdot d$ the irradiated volume.

Apparatus

1 X-ray apparatus with X-ray tube Mo and goniometer or 1 X-ray apparatus with X-ray tube Cu and goniometer	554 811
1 X-ray energy detector	559 938
1 Target set of alloys	554 848
1 Target set for K-lines fluorescence	554 844
1 Target set for L-lines fluorescence	554 846
1 Sensor-CASSY	524 010
1 MCA Box	524 058
1 CASSY Lab	524 200
1 BNC cable, 1 m	501 02
1 PC with Windows 98/2000 or higher version	

Using this information, the mass ratio C_i of the element number i in the alloy is

$$C_i = \frac{n_i \cdot A_i}{\sum_i n_i \cdot A_i} = \frac{\rho_i \cdot \dfrac{H_i}{H_{0i}}}{\sum_i \rho_i \cdot \dfrac{H_i}{H_{0i}}} \cdot \qquad (I)$$

The X-ray apparatus fulfils all regulations on the design of an X-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (NW 807 / 97 Rö).

The built-in protective and shielding fixtures reduce the dose rate outside the X-ray apparatus to less than 1 µSv/h, which is of the order of magnitude of the natural background radiation.

■ Before putting the X-ray apparatus into operation, inspect it for damage and check whether the high voltage is switched off when the sliding doors are opened (see instruction sheet of the X-ray apparatus).

■ Protect the X-ray apparatus against access by unauthorized persons.

Avoid overheating of the anode in the X-ray tube.

■ When switching the X-ray apparatus on, check whether the ventilator in the tube chamber starts rotating.

The goniometer is positioned solely by means of electric stepper motors.

■ Do not block the target arm and sensor arm and do not use force to move them.
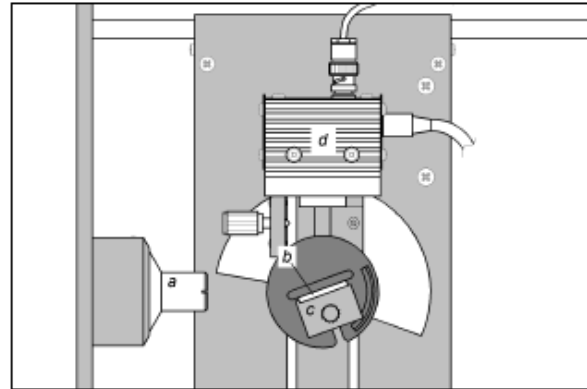

Fig. 1: Experiment setup: a – collimator, b – target, c – target stage, d – detector.

Setup

Fig. 1 shows the experiment setup.

- Guide the connection cable for the table-top power supply through the empty channel of the x-ray apparatus and connect it to the mini-DIN socket of the x-ray energy detector.

- Secure the sensor holder with the mounted x-ray energy detector in the goniometer sensor arm.

- Connect the signal output of the x-ray energy detector to the BNC socket SIGNAL IN of the x-ray apparatus by means of the BNC cable included.

- Feed enough connection cable through to make complete movement of the sensor arm possible.

- Press the SENSOR button and set the sensor angle with the rotary adjuster ADJUST manually to 90°.

- Connect Sensor-CASSY to the computer and connect the MCA box.

- Connect the SIGNAL OUT output in the connection panel of the x-ray apparatus to the MCA box by means of the BNC cable.

- Set the distances between the slit aperture of the collimator and the axis of rotation as well as between the axis of rotation and the window of the x-ray energy detector both to 5 to 6 cm.

- Place target 3 (brass containing lead) from the target set of alloys onto the target table.

- Press the TARGET button and adjust the target angle manually using the rotary button ADJUST to 45°.

Carrying out the experiment

- Connect the table-top power supply to the mains (after approx. 2 minutes the LED of the x-ray energy detector will glow "green" indicating that it is ready for use).

- Call CASSY Lab, and select the measuring parameters "Multichannel Measurement, 512 Channels, Negative Pulses, Gain 2.5, Measuring Time 180 s".

- Select the tube high voltage $U = 35$ kV and the emission current $I = 1$ mA, and switch the high voltage on.

- Start recording the spectrum with ⊙ or with the F9 key.

- Finally, record the spectra for the targets Cu, Zn and Pb from the target sets for K lines and L lines fluorescence as reference spectra.
- Save the entire measurement using an appropriate name.

Measuring example

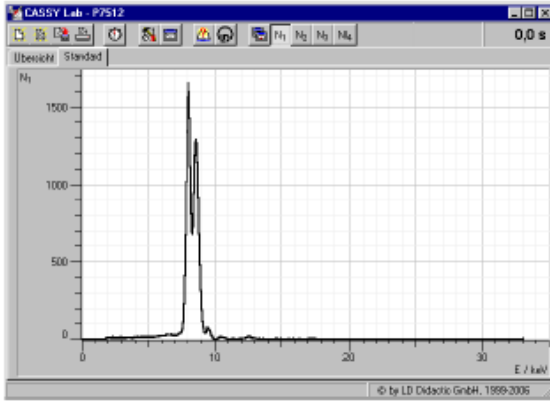The recorded spectra are displayed in Fig. 2a-d.
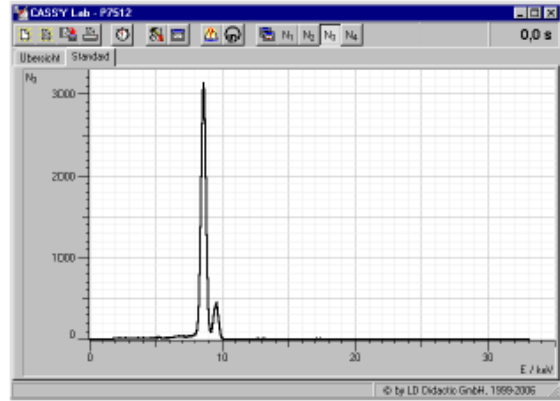

Fig. 2a Brass fluorescence spectrum.

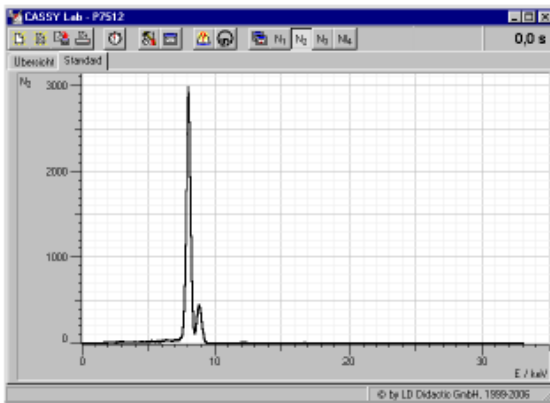Fig. 2c Zinc fluorescence spectrum (reference spectrum).

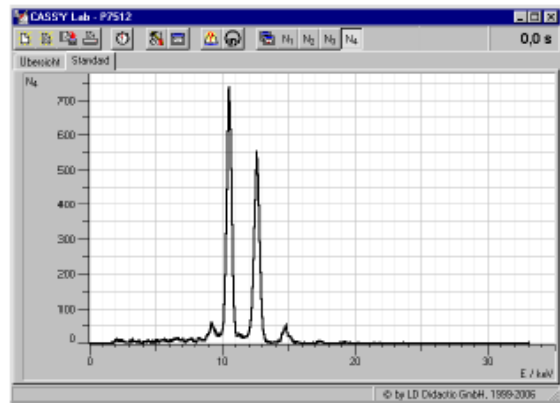Fig. 2b Copper fluorescence spectrum (reference spectrum).

Fig. 2d Lead fluorescence spectrum (reference spectrum).

Evaluation

a) Energy calibration of the spectra

The energy calibration is carried out for the spectra of copper and lead from the reference spectra.

- Open the "Energy Calibration" dialog window with the shortcut Alt+E, select "Global Energy Calibration" and enter the energies of the Cu Kα line (8.04 keV) and the Pb Lα line (10.56 keV).
- Select the menu item "Other Evaluations" → "Calculate Peak Center" in the pop-up menu of the diagram window, mark the Cu Kα line, and enter the result in the "Energy Calibration" dialog window.
- Then determine and enter the centre for the Pb Lα line.

b) Identifying the lines in the brass spectrum

For identifying and labelling the lines (see fig. 3a)

- In the pop-up menu of the diagram window select the item "X-ray Energies".
- Click on the element symbol Cu and select the button "Adopt".
- Finally plot the lines for zinc (Zn) and lead (Pb).

It is apparent that the second largest peak in the spectrum consists of two non-resolved lines: Zn Kα and Cu Kβ. The Cu Kα line is in part superimposed with the Zn Kα line.
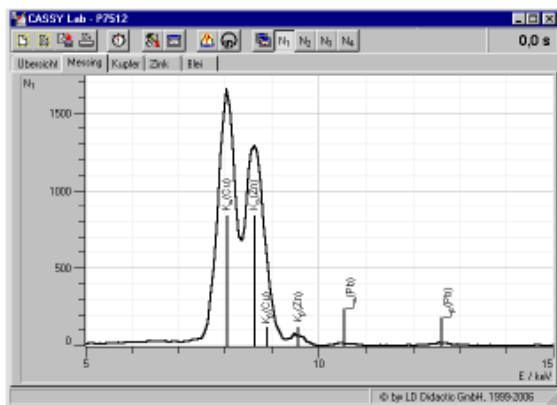

Fig. 3a Fluorescence spectrum of the brass sample with the identified lines of the alloy components.

c) Resolution of the superimposed lines in the fluorescence spectrum of the brass sample

The mass ratios of the alloy components are calculated by comparing the heights of the strongest lines in the brass fluorescence spectrum and the reference spectra. These lines are: the Kα of copper, the Kα of zinc and the Lα of lead.

For the determination of the heights of the Cu Kα and the Zn Kα, the brass fluorescence spectrum must be resolved in the energy range from 7.5 keV to 9.1 keV. For this the spectrum in this range is fitted with three Gaussian curves of equal width at the known energies of the Cu Kα line (E = 8.04 keV), the Cu Kβ line (8.91 keV) and the Zn Kα line (8.64 keV).

- In the pop-up menu of the diagram window select the item "Other Evaluations" → "Gaussians of specified energy".
- Mark measuring points in the energy range 7.5 keV to 9.1 keV (without the Pb LI line).

The result is a fitted contour of the fluorescence spectrum (see fig. 3b). The final values of the parameters should be taken from the status line and inserted in a table (H in Tab. 1).
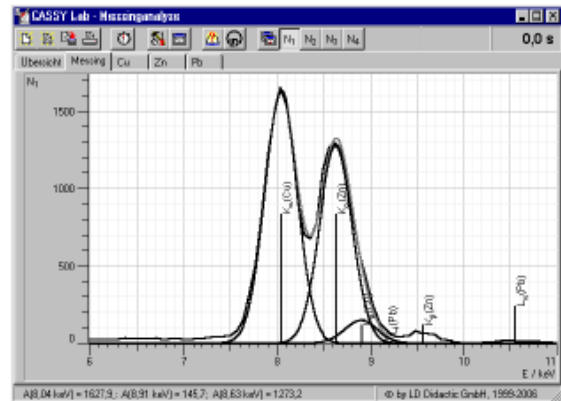

Fig. 3b Result of fitting the brass fluorescence spectrum with three Gaussian curves (lines) of given energies.

To determine the height of the Lα line for lead in the fluorescence spectrum of the brass sample, a Gaussian curve is fitted to it:

- In the pop-up menu of the diagram window select the item "Other Evaluations" → "Fit Gaussians" (Alt+G).
- Mark the Pb Lα line (energy range from 10 keV to 11.5 keV).
- Insert the result (A_1) in Tab. 1 (H).

d) Determination of the line heights in the reference spectra

- In the pop-up menu of the diagram window select the item "Other Evaluations" → "Fit Gaussians" (Alt+G).
- Mark the Cu Kα line in the reference spectrum.
- Insert the result (A_1) in Tab. 1 (H_0).
- Finally determine the heights of the Zn Kα line and the Pb Lα line in their reference spectra and insert the values in Tab. 1.

e) Determination of the mass ratios

The mass ratios of the alloy components are determined according to Eq. (I). The values of the peak heights in the brass fluorescence spectrum (H) and in the reference spectra (H_0) are stated in Tab. 1.

Tab. 1: Determination of the mass ratios C of the alloy components of the brass sample.

Element	ρ, g/cm^3	Line	H	H_0	$\rho \cdot H/H_0$	C, %
Copper	8,96	Kα	1628	2959	4,91	61,3
Zinc	7,10	Kα	1273	3132	2,79	35,9
Lead	11,34	Lα	14,6	711	0,23	2,9

Result

The determined mass ratios of the alloy components in the brass sample correspond well to the known chemical composition (CuZn39Pb3).

Tab. 2: Comparison of the experimental values with the stated mass ratios of the alloy components.

Element	Stated	Experimental
Copper	58%	61,3%
Zinc	39%	35,9%
Lead	3%	2,9%

Additional information

The example of the copper-zinc alloy (brass) shows how the *secondary fluorescence* modifies the spectrum shape. During the irradiation of such a sample with x-ray photons, the K lines of both copper and zinc are excited. But because the Kβ line of zinc (E = 9.57 keV) lies above the K edge of copper (E = 8.99 keV), it can also "secondarily" excite the copper K lines.

Therefore, in the fluorescence radiation emitted, the intensity of the copper lines is higher at the cost of the Zn Kβ line, and the ratio of the Zn Kα and the Kβ lines does not correspond to the ratio found in pure zinc. For this reason, the mass ratio of the alloy components determined from the Kα lines indicates a slightly too high proportion of copper.
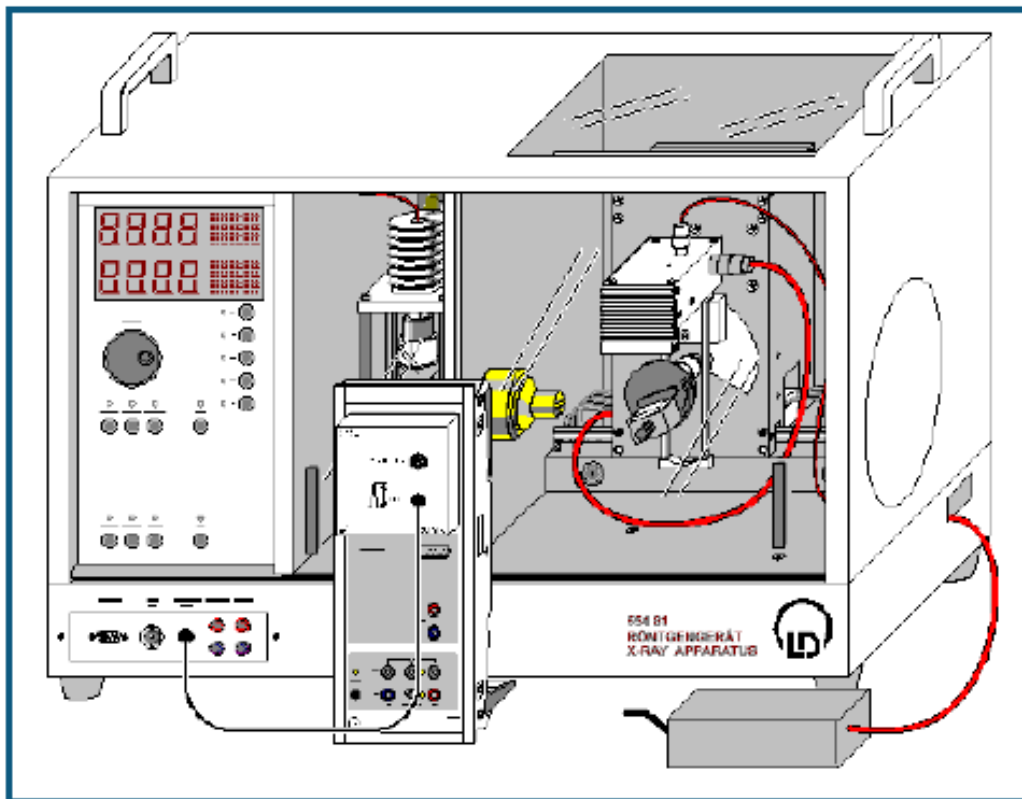
Solid-state physics

Applied solid-state physics
X-ray fluorescence analysis

Determination of the chemical composition of a brass sample by X-ray fluorescence analysis

Description from CASSY Lab 2

For loading examples and settings, please use the CASSY Lab 2 help.

Determination of the chemical composition of a brass sample (x-ray fluorescence)

can also be carried out with Pocket-CASSY

Safety notes

The X-ray apparatus fulfils all regulations on the design of an X-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (BfS 05/07 V/Sch RöV or NW 807 / 97 Rö).

The built-in protective and shielding fixtures reduce the dose rate outside the X-ray apparatus to less than 1 µSv/h, which is of the order of magnitude of the natural background radiation.

- Before putting the X-ray apparatus into operation, inspect it for damage and check whether the voltage is switched off when the sliding doors are opened (See instruction sheet of the X-ray apparatus).
- Protect the X-ray apparatus against access by unauthorized persons.

Avoid overheating of the anode in the X-ray tube.

- When switching the X-ray apparatus on, check whether the ventilator in the tube chamber starts rotating.

The goniometer is positioned solely by means of electric stepper motors.

- Do not block the target arm and the sensor arm of the goniometer and do not use force to move them.

When handling heavy metals or allergen substances from the target set, observe their operating instructions.

Experiment description

In this experiment, the quantitative analysis of the chemical composition of a brass sample containing lead is carried out. The components in this alloy were already identified in the experiment Non-destructive analysis of the chemical composition.

For calculating the mass ratios, the fact is made use of that the height of a peak is proportional to the number n of radiating atoms. In the reference spectrum this number n_0 is determined by the density of the substance ρ, its atomic weight A, the radiated area S and the effective thickness d of the irradiated layer:

$n_0 = S \cdot d \cdot \rho / A$.

For the number of atoms of each type in the alloy, to first approximation the expression

$n = n_0 \cdot H/H_0 = V \cdot \rho/A \cdot H/H_0$

can be used. In this H and H_0 are the peak heights in the spectrum to be analyzed and in the reference spectrum is $V = S \cdot d$ the irradiated volume. Using this information, the mass ratio C_i of the element number i in the alloy is

$$C_i = \frac{n_i \cdot A_i}{\sum_j n_j \cdot A_j} = \frac{\rho_i \cdot \dfrac{H_i}{H_{0i}}}{\sum_j \rho_j \cdot \dfrac{H_{0j}}{H_{0j}}}$$

Equipment list

1	Sensor-CASSY	524 010 or 524 013
1	CASSY Lab 2	524 220
1	MCA box	524 058
1	X-ray apparatus with x-ray tube Mo	554 801 or 554 811
1	Target set of alloys	554 848
1	Target set for K-line fluorescence	554 844
1	Target set for L-line fluorescence	554 846
1	X-ray energy detector	559 938
1	HF cable, 1 m	501 02
1	PC with Windows XP/Vista/7/8	

Experiment setup (see drawing)

- Guide the connection cable for the table-top power supply through the empty channel of the x-ray apparatus and connect it to the mini-DIN socket of the x-ray energy detector.
- Secure the sensor holder with the mounted x-ray energy detector in the goniometer sensor arm
- Connect the signal output of the x-ray energy detector to the BNC socket SIGNAL IN of the x-ray apparatus by means of the BNC cable included
- Feed enough connection cable through to make complete movement of the sensor arm possible
- Press the SENSOR button and set the sensor angle with the rotary adjuster ADJUST manually to 90°
- Set the distances between the slit aperture of the collimator and the axis of rotation as well as between the axis of rotation and the window of the x-ray energy detector both to 5 to 6 cm
- Press the TARGET button and adjust the target angle manually using the rotary button ADJUST to 45°.
- Connect Sensor-CASSY to the computer and connect the MCA box
- Connect the SIGNAL OUT output in the connection panel of the x-ray apparatus to the MCA box by means of the BNC cable.

Carrying out the experiment

☐ Load settings

- Connect the table-top power supply to the mains (after approx. 2 min the LED will glow green and the x-ray energy detector will be ready for use)
- Place target 3 (brass containing lead) from the target set of alloys onto the target table
- Set the tube high voltage U = 35 kV, emission current I = 1.00 mA and switch the high voltage on
- Start the spectrum recording with ⏱
- Finally, record the spectra for the targets Cu, Zn and Pb from the target sets for K-lines and L-lines fluorescence as reference spectra

Energy calibration

The energy calibration is carried out using the spectra of copper and lead (reference spectra).

- Open in the Settings EA (right mouse button) the Energy calibration, select **Global for all spectra of this input** and enter on the right-hand side the energies of the Cu K_α-line (8.04 keV) and of the Pb L_α-line (10.56 keV).
- In the context menu of the diagram select Calculate peak center, mark the Cu K_α-line and enter the result in the left-hand side of the Energy calibration (e.g. with drag & drop from the status line)
- Then determine the center for the Pb L_α-line and also enter it on the left-hand side.
- Switch the display to energy (e.g. with Drag & Drop of E_A into the diagram)

Evaluation

For identifying and labeling the lines in the brass spectrum:

- In the context menu of the diagram select Set Marker → X-ray Energies → Fe
- Then enter the lines of zinc (Zn) and lead (Pb)

It becomes apparent that the second-highest peak in the spectrum consists of two lines which are not resolved: Zn K_α and Cu K_β. The Cu K_β-line is in part superimposed with the Zn K_α-line.

The mass ratios of the alloy components are calculated by comparing the heights of the strongest lines in the fluorescence spectrum of brass and the reference spectra. These lines are: the copper K_α, the zinc K_α and the lead L_α.

For the determination of the heights of the Cu K_α and the Zn K_α, the brass fluorescence spectrum must be resolved in the energy range from 7.5 keV to 9.1 keV. For this the spectrum in this range is fitted with three Gaussian curves of equal width at the known energies of the Cu K_α-line (E = 8.04 keV), the Cu K_β-line (8.91 keV) and the Zn K_α-line (8.64 keV). The best way to do this is using the fit Gaussians of specified Energies. When selecting the area, it must be observed that all the three required energy lines are contained in the area (do not include the Pb L_I-line).

The result is a fitted contour of the fluorescence spectrum. The determined heights H are found in the status line and are to be entered in the **Mass Proportion** diagram (e.g. by drag & drop) together with the densities ρ for Cu (ρ = 8.96 g/cm³), Zn (ρ = 7.10 g/cm³) and Pb (ρ = 11.34 g/cm³).

The same applies to the heights H_0 for the three reference spectra. When the three densities and the six heights have been entered, the three mass ratios are automatically calculated.

The determined mass ratios of the alloy components in the brass sample correspond well to the known chemical composition (CuZn39Pb3).

Element	Stated	Experimental
Copper	58 %	61.6 %
Zinc	39 %	35.6 %
Lead	3 %	2.9 %

Additional information

The example of the copper-zinc alloy (brass) shows how the secondary fluorescence modifies the spectrum shape. During the irradiation of such a sample with x-ray photons, the K-lines of both copper and zinc are excited. But because the K_β-line of zinc (E = 9.57 keV) lies above the K-edge of copper (E = 8.99 keV), it can also "secondarily" excite the copper L-lines.

Therefore, in the fluorescence radiation emitted, the intensity of the copper lines is higher at the cost of the Zn K_β-line, and the ratio of the Zn K_α and the K_β-lines does not correspond to the ratio found in pure zinc. For this reason, the mass ratio of the alloy components determined from the K_α-lines indicates a slightly too high proportion of copper.

Electron Diffraction

Pre Lab Questions:

1- Explain de Broglie's hypothesis and what is meant by de Broglie's wave?

2- Explain how the structure of crystalline solids can be studied using electron diffraction experiments?

3- Why is it possible to use a crystal as a diffraction grating for electron waves?

4- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➢ Prove the "de Broglie" hypothesis.

➢ Determine the lattice plane spacing of a graphite crystal.

➢ Calculate the experimental value of Plank's constant.

To achieve these aims:

▪ Measuring of radius of inner and outer rings as function of voltage.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

Diffraction of electrons in a polycrystalline lattice (Debye-Scherrer diffraction)

Objects of the experiment

- Determination of wavelength of the electrons
- Verification of the de Broglie's equation
- Determination of lattice plane spacings of graphite

Principles

Louis de Broglie suggested in 1924 that particles could have wave properties in addition to their familiar particle properties. He hypothesized that the wavelength of the particle is inversely proportional to its momentum:

$$\lambda = \frac{h}{p} \qquad (I)$$

λ: wavelength

h: Planck's constant

p: momentum

His conjecture was confirmed by the experiments of Clinton Davisson and Lester Germer on the diffraction of electrons at crystalline Nickel structures in 1927.

In the present experiment the wave character of electrons is demonstrated by their diffraction at a polycrystalline graphite lattice (Debye-Scherrer diffraction). In contrast to the experiment of Davisson and Germer where electron diffraction is observed in reflection this setup uses a transmission diffraction type similar to the one used by G.P. Thomson in 1928.

From the electrons emitted by the hot cathode a small beam is singled out through a pin diagram. After passing through a focusing electron-optical system the electrons are incident as sharply limited monochromatic beam on a polycrystalline graphite foil. The atoms of the graphite can be regarded as a space lattice which acts as a diffracting grating for the electrons. On the fluorescent screen appears a diffraction pattern of two concentric rings which are centred around the indiffracted electron beam (Fig. 1). The diameter of the concentric rings changes with the wavelength λ and thus with the accelerating voltage U as can be seen by the following considerations:
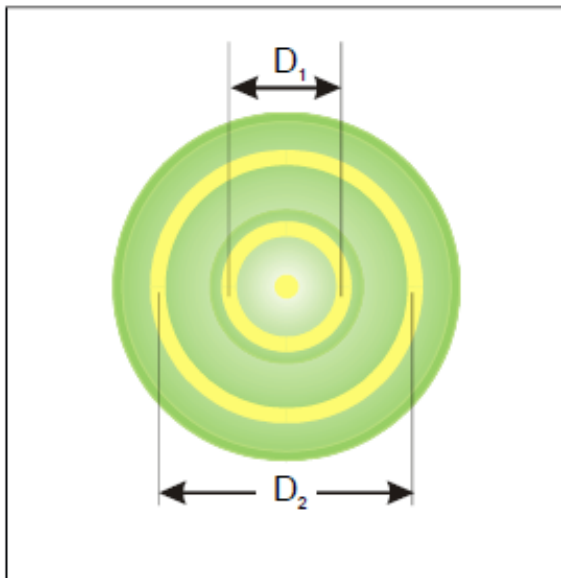

Fig. 1: Schematic representation of the observed ring pattern due to the diffraction of electrons on graphite. Two rings with diameters D_1 and D_2 are observed corresponding to the lattice plane spacings d_1 and d_2 (Fig. 3).

From energy equation for the electrons accelerated by the voltage U

$$e \cdot U = \frac{1}{2} m \cdot v^2 = \frac{p^2}{2 \cdot m} \qquad \text{(II)}$$

U: accelerating voltage

e: electron charge

m: mass of the particle

v: velocity of the particle

the momentum p can be derived as

$$p = m \cdot v = \sqrt{2 \cdot e \cdot m \cdot U} \qquad \text{(III)}$$

Substituting equation (III) in equation (I) gives for the wavelength:

$$\lambda = \frac{h}{\sqrt{2 \cdot m \cdot e \cdot U}} \qquad \text{(IV)}$$

In 1913, H. W. and W. L. Bragg realized that the regular arrangement of atoms in a single crystal can be understood as an array of lattice elements on parallel lattice planes. When we expose such a crystal lattice to monochromatic x-rays or mono-energetic electrons, and, additionally assuming that those have a wave nature, then each element in a lattice plane acts as a "scattering point", at which a spherical wavelet forms. According to Huygens' principle, these spherical wavelets are superposed to create a "reflected" wave front. In this model, the wavelength λ remains unchanged with respect to the "incident" wave front, and the radiation directions which are perpendicular to the two wave fronts fulfil the condition "angle of incidence = angle of reflection".

Constructive interference arises in the neighbouring rays reflected at the individual lattice planes when their path differences $\Delta = \Delta_1 + \Delta_2 = 2 \cdot d \cdot \sin \vartheta$ are integer multiples of the wavelength λ (Fig. 2):

$$2 \cdot d \cdot \sin \vartheta = n \cdot \lambda \qquad n = 1, 2, 3, \dots \qquad \text{(V)}$$

d: lattice plane spacing

ϑ: diffraction angle

This is the so called 'Bragg condition' and the corresponding diffraction angle ϑ is known as the glancing angle.

In this experiment a polycrystalline material is used as diffraction object. This corresponds to a large number of small single crystallites which are irregularly arranged in space. As a result there are always some crystals where the Bragg condition is satisfied for a given direction of incidence and wavelength. The reflections produced by these crystallites lie on a cones whose common axis is given by the direction of incidence. Concentric circles thus appear on a screen located perpendicularly to this axis. The lattice planes which are important for the electron diffraction pattern obtained with this setup possess the lattice plane spacings (Fig. 3):

$d_1 = 2.13 \cdot 10^{-10}$ m

$d_2 = 1.23 \cdot 10^{-10}$ m

From Fig. 4 we can deduce the relationship

$$\tan 2 \cdot \vartheta = \frac{D}{2 \cdot L} \qquad \text{(VI)}$$
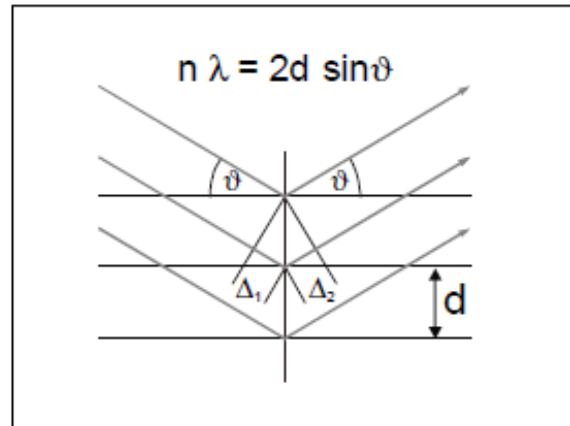

Fig. 2: Schematic representation of the Bragg condition.

If we approximate $\tan 2 \cdot \vartheta = \sin 2 \cdot \vartheta = 2 \cdot \sin \vartheta$ for small angles we obtain

$$2 \cdot \sin \vartheta = \frac{D}{2 \cdot L} \qquad \text{(VII)}$$

The substitution of equation (VII) in (V) leads in first order diffraction (n = 1) to

$$\lambda = d \cdot \frac{D}{2 \cdot L} \qquad \text{(VIII)}$$

D: ring diameter

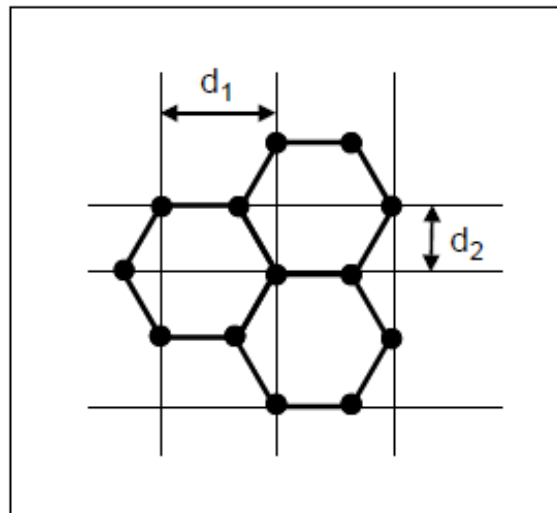L: distance between graphite and screen

d: lattice plane spacing

Fig. 3 Lattice plane spacings in graphite:
   $d_1 = 2.13 \cdot 10^{-10}$ m
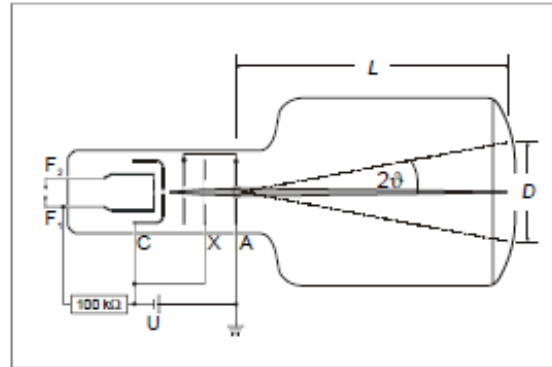 $d_2 = 1.23 \cdot 10^{-10}$ m

Apparatus

Fig. 4: Schematic sketch for determining the diffraction angle.
L = 13.5 cm (distance between graphite foil and screen),
D: diameter of a diffraction ring observed on the screen
ϑ: diffraction angle
For meaning of F_1, F_2, C, X and A see Fig. 5.

Due to equation (IV) the wavelength λ is determined by the accelerating voltage U. Combining the equation (IV) and equation (VIII) shows that the diameters D_1 and D_2 of the concentric rings change with the accelerating voltage U:

$$D = k \cdot \frac{1}{\sqrt{U}} \qquad (IX)$$

with

$$k = \frac{2 \cdot L \cdot h}{d \cdot \sqrt{2} \cdot m \cdot e} \qquad (X)$$

Measuring Diameters D_1 and D_2 as function of the accelerating voltage U allows thus to determine the lattice plane spacings d_1 and d_2.

Setup

The experimental setup (wiring diagram) is shown in Fig. 5.

- Connect the cathode heating sockets F1 and F2 of the tube stand to the output on the back of the high-voltage power supply 10 kV.
- Connect the sockets C (cathode cap) and X (focussing electrode) of the tube stand to the negative pole.
- Connect the socket A (anode) to the positive pole of the 5 kV/2 mA output of the high-voltage power supply 10 kV.
- Ground the positive pole on the high-voltage power supply 10 kV.

Safety notes

When the electron diffraction tube is operated at high voltages over 5 kV, X-rays are generated.

■ Do not operate the electron diffraction tube with high voltages over 5 keV.

The connection of the electron diffraction tube with grounded anode given in this instruction sheet requires a high-voltage enduring voltage source for the cathode heating.

■ Use the high-voltage power supply 10 kV (521 70) for supplying the electron diffraction tube with power.

Danger of implosion: the electron diffraction tube is a high-vacuum tube made of thin-walled glass.

■ Do not expose the electron diffraction tube to mechanical stress, and connect it only if it is mounted in the tube stand.

■ Treat the contact pins in the pin base with care, do not bend them, and be careful when inserting them in the tube stand.

The electron diffraction tube may be destroyed by voltages or currents that are too high:

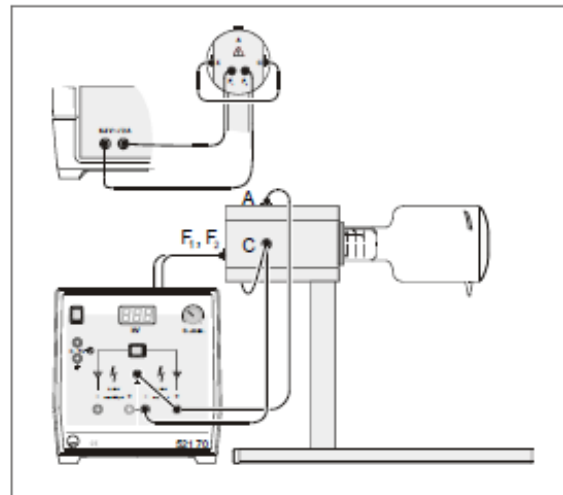■ Keep to the operating parameters given in the section on technical data.

Fig. 5: Experimental setup (wiring diagram) for observing the electron diffraction on graphite. Pin connection:
F_1, F_2: sockets for cathode heating
C: cathode cap
X: focusing electrode
A: anode (with polycrystalline graphite foil see Fig. 4)

Carrying out the experiment

- Apply an accelerating voltage U ≤ 5 kV and observe the diffraction pattern.

Hint: The direction of the electron beam can be influenced by means of a magnet which can be clamped on the neck of tube near the electron focusing system. To illuminate an another spot of the sample an adjustment of the magnet might be necessary if at least two diffraction rings cannot be seen perfectly in the diffraction pattern.

- Vary the accelerating voltage U between 3 kV and 5 kV in step of 0.5 kV and measure the diameter D_1 and D_2 of the diffraction rings on the screen (Fig. 1).
- Measure the distance between the graphite foil and the screen.

Measuring example

Table 1: Measured diameters D_1 and D_2 (average of 5 measurements) of the concentric diffraction rings as function of the accelerating voltage U.

$\dfrac{U}{kV}$	$\dfrac{D_1}{cm}$	$\dfrac{D_2}{cm}$
3.0	3.30	5.25
3.5	2.83	4.88
4.0	2.66	4.58
4.5	2.40	4.35
5.0	2.33	4.12

Distance between graphite foil and screen: L = 13.5 cm

Evaluation and results

a) Determination of wavelength of the electrons

From the measured values for D_1 and D_2 and the lattice plane spacings d_1 and d_2 the wavelength can be determined using equation (VIII). The result for D_1 and D_2 is summarized in Table 2 and Table 3, respectively.

Note: Rewriting equation (VIII) as

$$d = \lambda \cdot \frac{2 \cdot L}{D}$$

shows that the diameter D of the rings (Fig. 1) is inversely proportional to the lattice plane spacings d (Fig. 2). This information is necessary for the evaluation of the wavelength from the lattice plane spacings (here assumed as known) according equation (VIII). The lattice plane parameters are derived directly in part c) using equations (IX) and (X).

The dominant error in the measurement is the determination of the ring diameters D_1 and D_2. For an accuracy of reading about 2 mm the error is approximately 5% for the outer ring and approximately 10% for the inner ring.

Table 2: Measured diameter D_1 of the concentric diffraction rings as function of the accelerating voltage U. The wavelengths λ_1 and $\lambda_{1,theory}$ are determined by equation (VIII); and equation (IV), respectively.

$\dfrac{U}{kV}$	$\dfrac{D_1}{cm}$	$\dfrac{\lambda_1}{pm}$	$\dfrac{\lambda_{1,theory}}{pm}$
3.0	3.30	22.9	22.4
3.5	2.83	21.1	20.7
4.0	2.66	19.4	19.4
4.5	2.40	18.5	18.3
5.0	2.33	17.6	17.3

Table 3: Measured diameter D_2 of the concentric diffraction rings as function of the accelerating voltage U. The wavelengths λ_2 and $\lambda_{2,theory}$ are determined by equation (VIII); and equation (IV), respectively.

$\dfrac{U}{kV}$	$\dfrac{D_2}{cm}$	$\dfrac{\lambda_2}{pm}$	$\dfrac{\lambda_{2,theory}}{pm}$
3.0	5.25	22.6	22.4
3.5	4.88	21.0	20.7
4.0	4.58	19.7	19.4
4.5	4.35	18.6	18.3
5.0	4.12	17.5	17.3

b) Verification of the de Broglie's equation

The de Broglie relation (equation (I)) can be verified using

$e = 1.6021 \cdot 10^{-19}$ C

$m = 9.1091 \cdot 10^{-31}$ kg

$h = 6.6256 \cdot 10^{-34}$ J·s

in equation (IV). The results for the wavelengths determined by equation (IV) are $\lambda_{1,theory}$ and $\lambda_{2,theory}$. They are listed for the diameters D_1 and D_2 in Table 2 and Table 3, respectively. The values λ_1 and λ_2 determined from the diffraction pattern agree quite well with the theoretical values $\lambda_{1,theory}$ and $\lambda_{2,theory}$ due to the de Broglie relation.

c) Determination of lattice plane spacings of graphite

In Fig. 6 the ring diameters D_1 and D_2 are plotted versus $1/\sqrt{U}$. The slopes k_1 and k_2 are determined by linear fits through the origin according equation (IX) to the experimental data:

$k_1 = 1,578 \cdot m \sqrt{V}$

$k_2 = 2,729 \cdot m \sqrt{V}$

Resolving equation (X) for the lattice plane spacing d

$$d = \frac{2 \cdot L \cdot h}{k \cdot \sqrt{2 \cdot m \cdot e}}$$

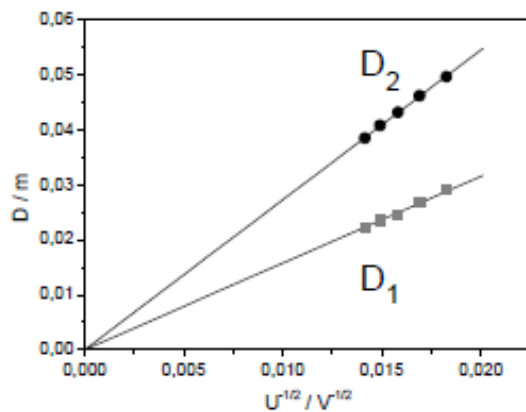Fig. 6: Ring diameters D_1 and D_2 as function of $1/\sqrt{U}$. The solid lines correspond to the linear fits with the slopes $k_1 = 1.578 \, m \sqrt{V}$ and $k_2 = 2.729 \, m \sqrt{V}$, respectively.

gives

$d_1 = 2.10 \cdot 10^{-10} \, m$

$d_2 = 1.21 \cdot 10^{-10} \, m$

which is within the error limits in accordance of the parameters depicted in Fig. 3.

Supplementary information

After the experiment of Davisson and Germer further experiments with particle wave effects due to particles confirmed the de Broglie relation and thus the wave-particle dualism. In 1930, for instance, O. Stern and I. Esterman succeeded in demonstrating the diffraction of hydrogen molecules and in 1931 they diffracted Helium atoms using a Lithium Fluoride crystal.

Experimental results which can be described by quantum theory only have the Planck constant h in their basic formula. In this experiment, for instance, the Planck's constant can be determined from equation (X) if the lattice spacings d_1 and d_2 of graphite are assumed to be known e.g. from x-ray structure analysis:

$$h = \frac{d \cdot k \cdot \sqrt{2 \cdot m \cdot e}}{2 \cdot L}$$

Using the values k_1 and k_2 obtained by the linear fit to experimental data (Fig. 6) gives

d_1: $h = 6.724 \cdot 10^{-34} \, J \cdot s$

d_2: $h = 6.717 \cdot 10^{-34} \, J \cdot s$

Literature: $h = 6.6256 \cdot 10^{-34} \, J \cdot s$

Hall Effect

Pre Lab Questions:

1- Explain briefly how an electromagnet works? Why do we use an iron core in our experiment?

2- Explain briefly without equations what is meant by Hall Effect?

3- Does the Hall Effect happen in both metals and semiconductors? Explain

4- Explain briefly what is meant by doping a semiconductor?

5- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➢ Study Hall Effect and determine Hall's constant.

➢ Calculate the concentration of carriers in a Ge Crystal.

➢ Calculate the Hall mobility of the carriers in the crystal.

To achieve these aims:

▪ Measuring of the Hall voltage as function of the magnetic field at a constant current.

▪ Measuring of the Hall voltage and the sample voltage as function of the current at a constant magnetic field.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

Hall effect in n-germanium with Cobra4 Mobile-Link

Related topics

Semiconductor, band theory, forbidden zone, intrinsic conduction, extrinsic conduction, valency band, conduction band, Lorentz force, magneto resistance, Neyer-Neldel rule.

Principle

The resistance and Hall voltage are measured on a rectangular strip of germanium as a function of the temperature and of the magnetic field. From the results obtained the energy gap, specific conductivity, type of charge carrier and the carrier mobility are determined.

Equipment

1	Cobra4 Mobile-Link set, incl. rechargeable batteries, SD memory card, USB cable and software "measure"	12620-55
1	Cobra4 Sensor Tesla, magnetic field strength, resolution max. ±0.01 mT	12652-00
1	Hall effect module,	11801-00
1	Hall effect, n-Ge, carrier board	11802-01
2	Coil, 600 turns	06514-01
1	Iron core, U-shaped, laminated	06501-00
1	Pole pieces, plane, 30x30x48 mm, 2	06489-00
1	Hall probe, tangent., prot. cap	13610-02
1	Power supply 0-12 V DC/6 V, 12 V AC	13505-93
1	Tripod base PHYWE	02002-55
1	Support rod PHYWE, square, l = 250 mm	02025-55
1	Right angle clamp PHYWE	02040-55
3	Connecting cord, l = 500 mm, red	07361-01
2	Connecting cord, l = 500 mm, blue	07361-04
2	Connecting cord, l = 750 mm, black	07362-05
1	Digital multimeter 2010	07128-00

Fig. 1: Experimental setup.

Task

1. At constant room temperature and with a uniform magnetic field measure the Hall voltage as a function of the control current and plot the values on a graph (measurement without compensation for error voltage).

Fig. 2: Hall effect on a rectangular specimen. The polarity of the Hall voltage indicated is for negative charge carriers.

2. At room temperature and with a constant control current, measure the voltage across the specimen as a function of the magnetic flux density B.

3. Keeping the control current constant measure the voltage across the specimen as a function of temperature. From the readings taken, calculate the energy gap of germanium.

4. At room temperature measure the Hall Voltage U_H as a function of the magnetic flux density B. From the readings taken, determine the Hall coefficient R_H and the sign of the charge carriers. Also calculate the Hall mobility μ_H and the carrier density n.

5. Measure the Hall voltage U_H as a function of temperature at uniform magnetic flux density B, and plot the readings on a graph.
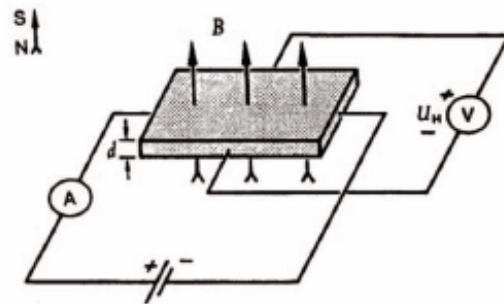
Setup and Procedure

The experimental setup is shown in Fig.1. The test piece on the board has to be put into the hall-effect-module via the guide-groove. The module is directly connected with the 12 V~ output of the power unit over the ac-input on the back-side of the module.

The plate has to be brought up to the magnet very carefully, so as not to damage the crystal in particular, avoid bending the plate.

The Hall voltage and the voltage across the sample are measured with a multimeter. Therefore, use the sockets on the front-side of the module. The current and temperature can be easily read on the integrated display of the module.

The magnetic field has to be measured with the Cobra4 Sensor-Unit Tesla via a hall probe, which can be directly put into the groove in the module as shown in Fig.1. So you can be sure that the magnetic flux is measured directly on the Ge-sample.

1. Set the magnetic field to a value of 250 mT by changing the voltage and current on the power supply. Connect the multimeter to the sockets of the hall voltage (U_H) on the front-side of the module. Set the display on the module into the "current-mode". Determine the hall voltage as a function of the current from -30 mA up to 30 mA in steps of nearly 5 mA.

   You will receive a typical measurement like in Fig.3.

2. Set the control current to 30 mA. Connect the multimeter to the sockets of the sample voltage on the front-side of the module. Determine the sample voltage as a function of the positive magnetic induction B up to 300 mT.

   You will get a typical graph as shown in Fig.4.

3. Be sure, that the display works in the temperature mode during the measurement. At the beginning, set the current to a value of 30 mA. The magnetic field is off. The current remains nearly constant during the measurement, but the voltage changes according to a change in temperature. Set the display in the temperature mode, now. Start the measurement by activating the heating coil with the "on/off"-knob on the backside of the module. Determine the change in voltage dependent on the change in temperature for a temperature range of room temperature to a maximum of 170°C.

You will receive a typical curve as shown in Fig.5.

4. Set the current to a value of 30 mA.

Connect the multimeter to the sockets of the hall voltage (U_H) on the front-side of the module. Determine the Hall voltage as a function of the magnetic induction. Start with -300 mT by changing the polarity of the coil-current and increase the magnetic induction in steps of nearly 20 mT. At zero point, you have to change the polarity. A typical measurement is shown in Fig.6.

5. Set the current to 30 mA and the magnetic induction to 300 mT.

Determine the Hall voltage as a function of the temperature.

Set the display in the temperature mode. Start the measurement by activating the heating coil with the "on/off"-knob on the backside of the module.

You will receive a curve like Fig.7.

Theory and evaluation

When a current-carrying conductor in the form of a rectangular strip is placed in a magnetic field with the lines of force at right angles to the current, a transverse e. m. f. – the so called Hall voltage – is set up across the strip.

This phenomenon is due to the Lorentz force: the charge carriers which give rise to the current flow through the specimen are deflected in the magnetic field B as a function of their sign and of their velocity v:
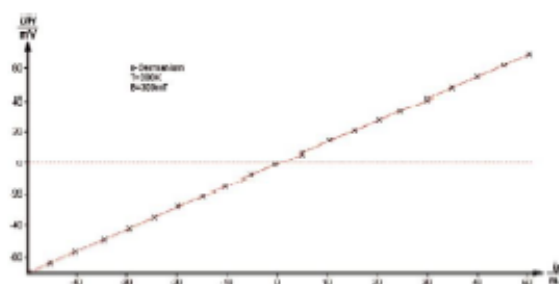
$$\vec{F} = e\,(\,\vec{v} \times B\,)$$

Fig. 4: Change of resistance as a function of the magnetic flux density.
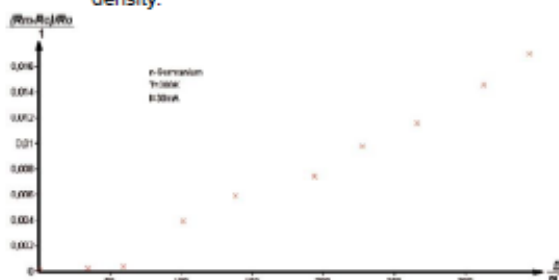
Fig. 5: The reciprocal specimen voltage as a function of the reciprocal absolute temperature (Since I was constant during the experiment, U−1 is approximately equal to s; the graph is therefore the same as a plot of the conductivity against the reciprocal temperature.)
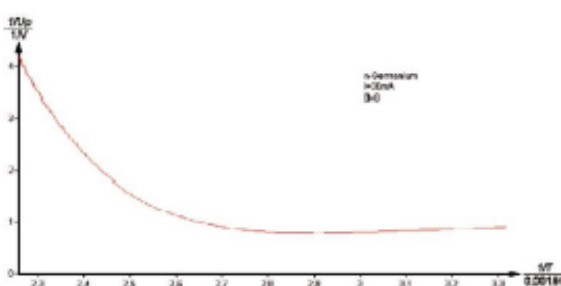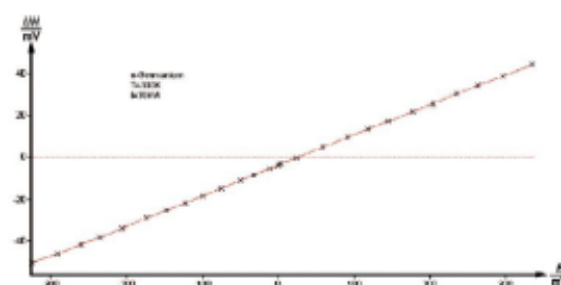
Fig. 6: Hall voltage as a function of the magnetic flux density.

(F = force acting on charge carriers, e = elementary charge).

Since negative and positive charge carriers have opposite directions of motion in the semiconductor, both are deflected in the same direction.

If the directions of the current and magnetic field are known, the polarity of the Hall voltage tells us whether the current is predominantly due to the drift of negative charges or to the drift of positive charges.

1. Fig. 3 shows that there is a linear relationship between the current I and the Hall voltage U_H:

$$U_H = \alpha \cdot I$$

(α = proportionality factor.)

2. The change of resistance of the specimen in a magnetic field is connected with a decrease of the mean free path of the charge carriers. Fig. 4 shows a non-linear, obviously quadratic change of resistance with increasing field strength.

3. For intrinsic conduction, the relationship between the conductivity σ and the absolute temperature T is:

$$\sigma = \sigma_0 \cdot \exp\left(-\frac{E_g}{2\,kT}\right)$$

where E_g is the energy gap between the valency and conduction bands, and k is Boltzmann's constant. A graph of $\log_e \sigma$ against $1/T$ will be linear with a slope of

$$b = -\frac{E_g}{2\,k}\,.$$

Hence E_g is obtained.

With the measured values in Fig. 5, the regression formulation

$$\ln \sigma = \ln \sigma_0 + \frac{E_g}{2\,k}\cdot T^{-1}$$

gives slope

$$b = -\frac{E_g}{2\,k} = -2.87\cdot 10^3\,\text{K}$$

with a standard deviation $s_b = \pm 0.3 \cdot 10^3\,\text{K}$.

(Since the experiment was performed with a constant current, σ can be replaced by U^{-1} [U = voltage across the specimen]).

Taking

$$k = 8.625 \cdot 10^{-5} \frac{eV}{K}$$

we obtain

$$E_g = b \cdot 2k = (0.50 \pm 0.04) \text{ eV}.$$

4. With the directions of control current und magnetic field illustrated in Fig. 2, the charge carriers which produce the current are deflected to the front edge of the specimen. If, therefore, the current is due mainly to electrons (as in the case of an n-doped specimen), the front edge becomes negatively charged. In the case of hole conduction (p-doped specimen) it becomes positively charged.

The conductivity σ_0, carrier mobility μ_H, and the carrier density n are all connected by the Hall coefficient R_H:

$$R_H = \frac{U_H}{B} \cdot \frac{d}{I}, \quad \mu_H = R_H \cdot \sigma_0$$

$$n = \frac{1}{e \cdot R_H}$$

Fig. 6 shows a linear relation between the Hall voltage and the magnetic flux density B. Using the values from Fig. 6, regression with the formulation

$$U_H = U_0 + b \cdot B$$

gives the slope $b = 0.144$ VT^{-1}, with the standard deviation $s_b + 0.004$ VT^{-1}.

The Hall coefficient R_H is then given by

$$R_H = \frac{U_H}{B} \cdot \frac{d}{I} = b \cdot \frac{d}{I}$$

Thus, if the thickness of specimen $d = 1 \cdot 10^{-3}$m and $I = 0.030$ A, then

$$R_H = 4.8 \cdot 10^{-3} \frac{m^3}{As}$$

with the standard deviation

$$s_{RH} = \pm 0.2 \cdot 10^{-3} \frac{m^3}{As} .$$

The conductivity at room temperature is calculated from the length l of the specimen, its cross-sectional area A and its resistance R_0 (cf. Experiment 2):

$$\sigma_0 = \frac{l}{R \cdot A} .$$

Thus if

$l = 0.02$ m, $R_0 = 37.3$ Ω, $A = 1 \cdot 10^{-5}$m^2, then

$$\sigma_0 = 53.6 \ \Omega^{-1} \ m^{-1}.$$

The Hall mobility μ_H of the charge carriers can now be determined from the expression

$$\mu_H = R_H \cdot \sigma_o$$

Using the same values above, this gives

$$\mu_H = (0.257 \pm 0.005)\ \frac{m^2}{Vs}\ .$$

The electron concentration n of n-doped specimen is given by

$$n = \frac{1}{e \cdot R_H}\ .$$

Taking e = elementary charge = $1.602 \cdot 10^{-19}$ As, we obtain

$$n = 13.0 \cdot 10^{20}\ m^{-3}.$$

5. Fig. 7 shows that the Hall voltage decreases with increasing temperature. Since the experiment was performed with a constant current, it can be assumed that the increase of charge carriers (transition from extrinsic to intrinsic conduction) with the associated reduction of the drift velocity v is responsible for this.

(The same current for a higher number of charge carriers means a lower drift velocity). The drift velocity is in turn related to the Hall voltage by the Lorentz force.

Note

For the sake of simplicity, only the magnitude of the Hall volt-age and Hall coefficient has been used here. These values are usually given a negative sign in the case of electron conduction.

Hall effect in p-germanium with Cobra4 Mobile-Link

Related Topics

Semiconductor, band theory, forbidden zone, intrinsic conductivity, extrinsic conductivity, valence band, conduction band, Lorentz force, magnetic resistance, mobility, conductivity, band spacing, Hall coefficient.

Principle

The resistivity and Hall voltage of a rectangular germanium sample are measured as a function of temperature and magnetic field. The band spacing, the specific conductivity, the type of charge carrier and the mobility of the charge carriers are determined from the measurements.

Equipment

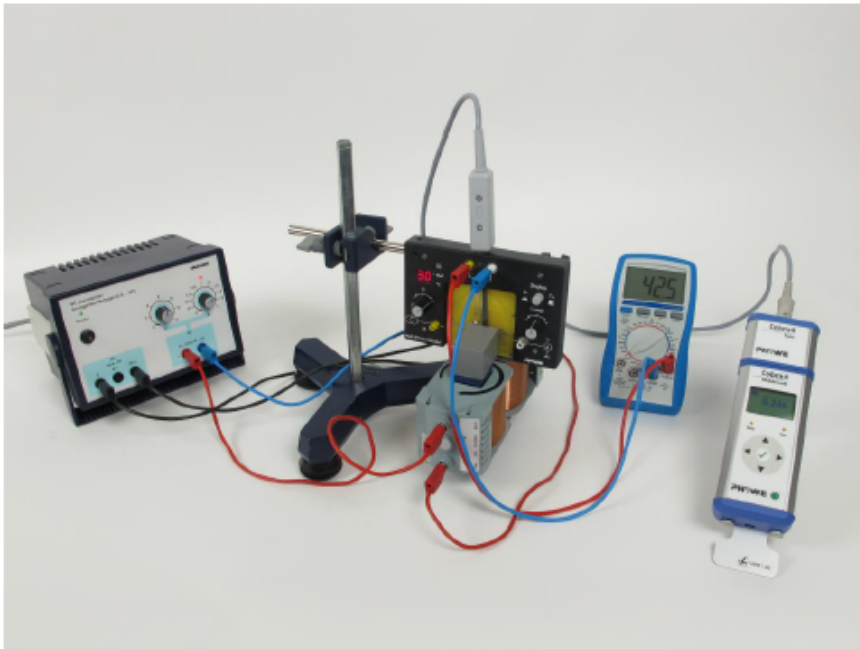|		
1	Cobra4 Mobile-Link set, incl. rechargeable batteries, SD memory card, USB cable and software "measure"	12620-55
1	Cobra4 Sensor Tesla, magnetic field strength, resolution max. ±0.01 mT	12652-00
1	Hall effect module,	11801-00
1	Hall effect, p-Ge, carrier board	11805-01
2	Coil, 600 turns	06514-01
1	Iron core, U-shaped, laminated	06501-00
1	Pole pieces, plane, 30x30x48 mm, 2	06489-00
1	Hall probe, tangent., prot. cap	13610-02
1	Power supply 0-12 V DC/6 V, 12 V AC	13505-93
1	Tripod base PHYWE	02002-55
1	Support rod PHYWE, square, l = 250 mm	02025-55
1	Right angle clamp PHYWE	02040-55
3	Connecting cord, l = 500 mm, red	07361-01
2	Connecting cord, l = 500 mm, blue	07361-04
2	Connecting cord, l = 750 mm, black	07362-05
1	Digital multimeter 2010	07128-00

Fig. 1:   Experimental setup.

Task

1. The Hall voltage is measured at room temperature and constant magnetic field as a function of the control current and plotted on a graph (measurement without compensation for defect voltage).

2. The voltage across the sample is measured at room temperature and constant control current as a function of the magnetic induction B.

3. The voltage across the sample is measured at constant control current as a function of the temperature. The band spacing of germanium is calculated from the measurements.
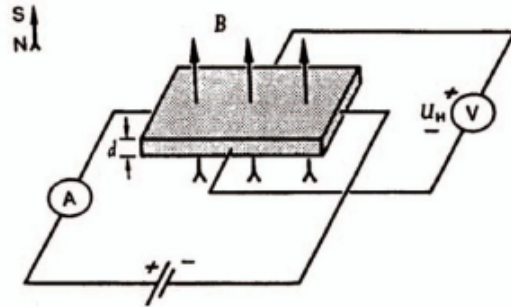

Fig. 2: Hall effect in sample of rectangular section. The polarity sign of the Hall voltage shown applies when the carriers are negatively charged.

4. The Hall voltage U_H is measured as a function of the magnetic induction B, at room temperature. The sign of the charge carriers and the Hall constant R_H together with the Hall mobility μH and the carrier concentration p are calculated from the measurements.

5. The Hall voltage U_H is measured as a function of temperature at constant magnetic induction B and the values are plotted on a graph.

Set-up and Procedure

The experimental set-up is shown in Fig.1. The test piece on the board has to be put into the hall-effect-module via the guide-groove. The module is directly connected with the 12 V~ output of the power unit over the ac-input on the backside of the module.

The plate has to be brought up to the magnet very carefully, so as not to damage the crystal in particular, avoid bending the plate.

The Hall voltage and the voltage across the sample are measured with a multimeter. Therefore, use the sockets on the front-side of the module. The current and temperature can be easily read on the integrated display of the module.

The magnetic field has to be measured with the Cobra4 Sensor-Unit Tesla via a hall probe, which can be directly put into the groove in the module as shown in Fig. 1. So you can be sure that the magnetic flux is measured directly on the Ge-sample.

Task 1

Set the magnetic field to a value of 250 mT by changing the voltage and current on the power supply. Connect the multimeter to the sockets of the hall voltage (U_H) on the front-side of the module. Set the display on the module into the "current-mode". Determine the hall voltage as a function of the current from -30 mA up to 30 mA in steps of nearly 5 mA. You will receive a typical measurement like in Fig. 3.

Task 2

Set the control current to 30 mA. Connect the multimeter to the sockets of the sample voltage on the front-side of the module. Determine the sample voltage as a function of the positive magnetic induction B up to 300 mT. You will get a typical graph as shown in Fig. 4.

Task 3

Be sure, that the display works in the temperature mode during the measurement. At the beginning, set the current to a value of 30 mA. The magnetic field is off. The current remains nearly constant during the measurement, but the voltage changes according to a change in temperature. Set the display in the temperature mode, now. Start the measurement by activating the heating coil with the "on/off"-knob on the backside of the module. Determine the change in voltage dependent on the change in temperature for a temperature range of room temperature to a maximum of 140°C. You will receive a typical curve as shown in Fig. 5.

Task 4

Set the current to a value of 30 mA. Connect the multimeter to the sockets of the hall voltage (U_H) on the front-side of the module. Determine the Hall voltage as a function of the magnetic induction. Start with - 300 mT by changing the polarity of the coil-current and increase the magnetic induction in steps of nearly 20 mT. At zero point, you have to change the polarity. A typical measurement is shown in Fig. 6.

Task 5:

Set the current to 30 mA and the magnetic induction to 300 mT. Determine the Hall voltage as a function of the temperature. Set the display in the temperature mode. Start the measurement by activating the heating coil with the "on/off"- knob on the backside of the module. You will receive a curve like Fig. 7.

Theory and evaluation

If a current I flows through a conducting strip of rectangular section and if the strip is traversed by a magnetic field at right angles to the direction of the current, a voltage – the so-called Hall voltage – is produced between two superposed points on opposite sides of the strip.

This phenomenon arises from the Lorentz force: the charge carriers giving rise to the current flowing through the sample are deflected in the magnetic field B as a function of their sign and their velocity v:

$$\vec{F} = e\,(\vec{v} \times B)$$

(F = force acting on charge carriers, e = elementary charge).

Since negative and positive charge carriers in semiconductors move in opposite directions, they are deflected in the same direction.

The type of charge carrier causing the flow of current can therefore be determined from the polarity of the Hall voltage, knowing the direction of the current and that of the magnetic field.

Task 1

Fig. 3 shows that there is a linear relationship between the current I and the Hall voltage U_H:

$$U_H = \alpha \cdot I$$
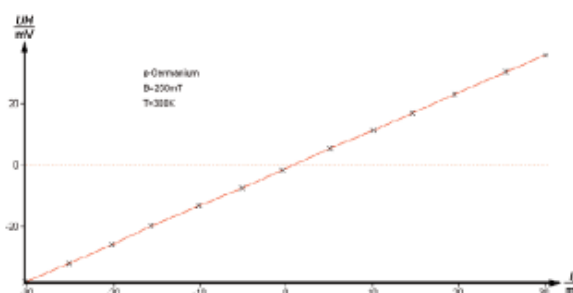
where α = proportionality factor.

Fig. 3: Hall voltage as a function of current.

Task 2

The change in resistance of the sample due to the magnetic field is associated with a reduction in the mean free path of the charge carriers. Fig. 4 shows the non-linear, clearly quadratic, change in resistance as the field strength increases.
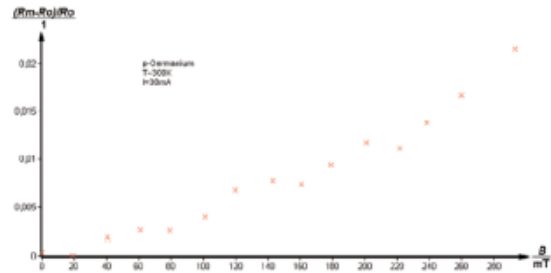

Fig. 4: Change of resistance as a function of magnetic induction.

Task 3

In the region of intrinsic conductivity, we have

$$\sigma = \sigma_0 \cdot \exp\left(-\frac{E_g}{2\,kT}\right)$$

where σ = conductivity, E_g = energy of bandgap, k = Boltzmann constant, T = absolute temperature.

If the logarithm of the conductivity is plotted against T^{-1} a straight line is obtained with a slope from which E_g can be determined. From the measured values used in Fig. 5, the slope of the regression line

$$\ln \sigma = \ln \sigma_0 + \frac{E_g}{2\,k} \cdot T^{-1}$$

is

$$b = -\frac{E_g}{2\,k} = -4.18 \cdot 10^3\,\text{K}$$

with a standard deviation $s_b = \cdot \pm 0.07 \cdot 10^3$ K. (Since the measurements were made with a constant current, we can put $s \sim U^{-1}$, where U is the voltage across the sample.)

$$k = 8.625 \cdot 10^{-5}\,\frac{\text{eV}}{\text{K}}$$

Since

$$E_g = b \cdot 2k = (0.72 \pm 0.03)\,\text{eV}.$$
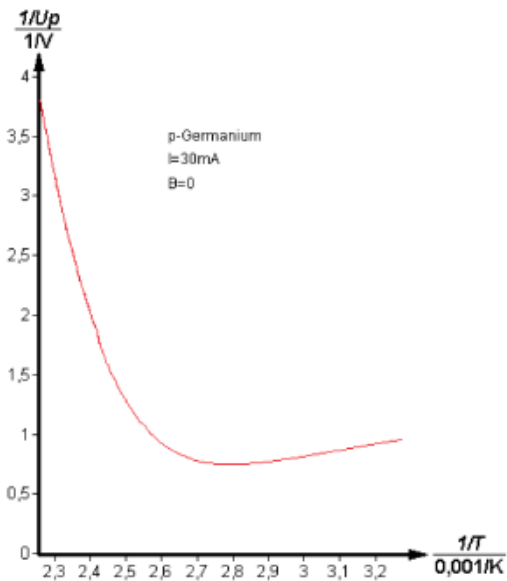
we get

Fig. 5: Reciprocal sample voltage plotted as a function of reciprocal absolute temperature. (Since I was constant during the measurement, $U^{-1} \sim \sigma$ and the graph is therefore equivalent to a plot of conductivity against reciprocal temperature).

Task 4

With the directions of control current and magnetic field shown in Fig. 2, the charge carriers giving rise to the current in the sample are deflected towards the front edge of the sample. Therefore, if (in an n-doped probe) electrons are the predominant charge carriers, the front edge will become negative, and, with hole conduction in a p-doped sample, positive.

The conductivity s0, the chargecarrier mobility μ_H, and the charge-carrier concentration p are related through the Hall constant R_H:

$$R_H = \frac{U_H}{B} \cdot \frac{d}{I}, \quad \mu_H = R_H \cdot \sigma_0$$

$$p = \frac{1}{e \cdot R_H}$$

Fig. 6 shows a linear connection between Hall voltage and B field. With the values used in Fig. 6, the regression line with the formula

$$U_H = U_0 + b \cdot B$$

has a slope b = 0.125 VT^{-1}, with a standard deviation $s_b \cdot \pm 0.003$ VT^{-1}.

The Hall constant R_H thus becomes, according to

$$R_H = \frac{U_H}{B} \cdot \frac{d}{I} = b \cdot \frac{d}{I}$$

Fig. 6: Hall voltage as a function of magnetic induction.

where the sample thickness d = 1 · 10^{-3}m and I = 0.030 A,

$$R_H = 4.17 \cdot 10^{-3} \frac{m^3}{As}$$

with the standard deviation

$$s_{RH} = 0.08 \cdot 10^{-3} \frac{m^3}{As}.$$

The conductivity at room temperature is calculated from the sample length l, the sample cross-section A and the sample resistance R_0 (cf. 2) as follows:

$$\sigma_0 = \frac{l}{R \cdot A}.$$

With the measured values

$$l = 0.02 \text{ m}, R_0 = 35.0 \, \Omega, A = 1 \cdot 10^{-5} m^2$$

we have

σ_0 = 57.14 Ω^{-1}m^{-1}.

The Hall mobility μ_H of the charge carriers can now be determined from

$$\mu_H = R_H \cdot \sigma_0$$

Using the measurements given above, we get:

$$\mu_H = (0.238 \pm 0.005) \frac{m^2}{Vs}.$$

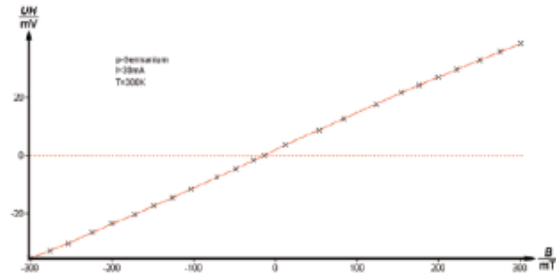The hole concentration p of p-doped samples is calculated from

$$p = \frac{1}{e \cdot R_H}$$

Using the value of the elementary charge

$e = 1.602 \cdot 10^{-19}$ As

we obtain

$p = 14.9 \cdot 10^{20}$ m^{-3}.

Task 5

Fig. 7 shows first a decrease in Hall voltage with rising temperature. Since the measurements were made with constant current, it is to be assumed that this is attributable to an increase in the number of charge carriers (transition from extrinsic conduction to intrinsic conduction) and the associated reduction in drift velocity v.

(Equal currents with increased numbers of charge carriers imply reduced drift velocity). The drift velocity in its turn is connected with the Hall voltage through the Lorentz force.

Fig. 7: Hall voltage as a function of temperature.

The current in the crystal is made up of both electron currents and hole currents

$$I = A \cdot e \, (v_n \cdot n + v_p \cdot p).$$

Since in the instrinsic velocity range the concentrations of holes p and of electrons n are approximately equal, those charge carriers will in the end make the greater contribution to the Hall effect which have the greater velocity or

(since $v = \mu \cdot E$) the greater mobility.

Fig. 7 shows accordingly the reversal of sign of the Hall voltage, typical of p-type materials, above a particular temperature.
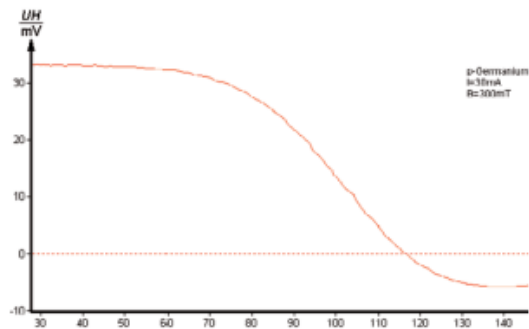
The Effect of temperature on metals and semiconductor resistance

Pre Lab Questions:

1- What are the basic new ideas that the quantum free electron model introduced regarding the electrical resistivity of metals?

2- Explain the band theory of semiconductors in particular?

3- According to this theory, what would be the effect of temperature on the conductivity of a semiconductor?

4- What is your goal for the experiment and how are you going to achieve it?

<u>The aims of this experiment are:</u>

<u>For a metal:</u>

- ➢ Study the effect of temperature on metal resistance.
- ➢ Determine the temperature coefficient of resistance of platinum.

<u>For a semiconductor:</u>

- ➢ Study the behavior of the resistance of a semiconductor at high temperature.
- ➢ Determine the energy gap of germanium.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment

sheets for **both** metal and semiconductor in <u>different files</u>!

<u>Finally:</u>

Discus your results!

Band gap of germanium

Related topics

Semiconductor, band theory, forbidden band, intrinsic conduction, extrinsic conduction, impurity depletion, valence band, conduction band.

Principle

The conductivity of a germanium testpiece is measured as a function of temperature. The energy gap is determined from the measured values.

Equipment

Hall effect module,	11801.00	1
Hall effect, undot.-Ge, carrier board	11807.01	1
Power supply 0-12 V DC/6 V, 12 V AC	13505.93	1
Tripod base -PASS-	02002.55	1
Support rod -PASS-, square, $l = 250$ mm	02025.55	1
Right angle clamp -PASS-	02040.55	1
Digital multimeter	07134.00	1
Connecting cord, $l = 500$ mm, black	07361.05	2
Connecting cord, $l = 100$ mm, red	07359.01	1
Connecting cord, $l = 100$ mm, blue	07359.04	1

Tasks

1. The current and voltage are to be measured across a germanium test-piece as a function of temperature.

2. From the measurements, the conductivity σ is to be calculated and plotted against the reciprocal of the temperature T. A linear plot is obtained, from whose slope the energy gap of germanium can be determined.

Set-up and procedure

The experimental set-up is shown in Fig.1. The test piece on the board has to be put into the hall-effekt-modul via the guide-groove. The module is directly connected with the 12 V– output of the power unit over the ac-input on the backside of the module.

The voltage across the sample is measured with a multimeter. Therefore, use the two lower sockets on the front-side of the module. The current and temperature can be easily read on the integrated display of the module. Be sure, that the display works in the temperature mode during the measurement. You can change the mode with the "Display"-knob. At the beginning, set the current to a value of 5 mA. The current remains nearly constant during the measurement, but the voltage changes according to a change in temperature. Set the display in the temperature mode, now. Start the measurement by activating the heating coil with the "on/off"-knob on the backside of the module. Determine the change in voltage dependent on the change in temperature for a temperature range of room temperature to a maximum of 170°C.

You will receive a typical curve as shown in Fig.2.

Fig.1: Experimental set-up for the determination of the band gap of germanium

Theory and evaluation

The conductivity σ is defined as following:

$$\sigma = \frac{1}{\rho} = \frac{l \cdot I}{A \cdot U} \left[\frac{1}{\Omega m} \right]$$

with ρ = specific recistivity, l = length of test specimen, A = cross section, I = current, U = voltage.
(Dimensions of Ge-plate $20 \times 10 \times 1$ mm^3)

The conductivity of semiconductors is characteristically a function of temperature. Three ranges can be distinguished: at low temperatures we have **extrinsic conduction** (range I), i.e. as the temperature rises charge carriers are activated from the impurities. At moderate temperatures (range II we talk of **impurity depletion,** since a further temperatue rise no longer produces activition of impurities. At high temperatures (range III) it is **intrinsic conduction** which finally predominates (see Fig. 3). In this instance charge carriers are additionally transferred by thermal excitation from the valence band to the conduction band. The temperature dependence is in this case essentially described by an exponential function.

$$\sigma = \sigma_0 \cdot \exp{-\frac{E_g}{2kT}}$$

(E_g = energy gap, k = Boltzmann's constant, T = absolute temperature).

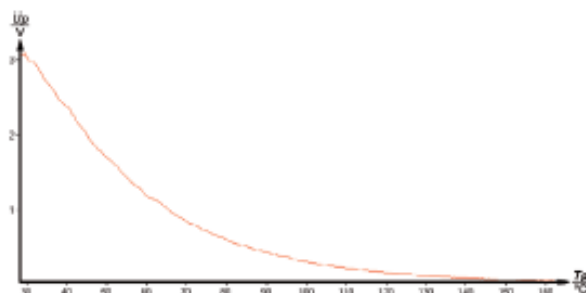The logarithm of this equation

$$\ln \sigma = \ln \sigma_0 - \frac{E_g}{2kT}$$

is with $y = \ln \sigma$ and $x = \frac{1}{T}$, a linear equation on the type $y = a + bx$, where

$$b = -\frac{E_g}{2k}$$

is slope of the straight line.

With the measured values from Fig. 2, the regression with the expression

$$\ln \sigma = \ln \sigma_0 + \frac{E_g}{2k} \cdot \frac{1}{T}$$

provides the slope $b = (4.05 \pm 0.06) \cdot 10^3$ K (Fig. 4).

With the Boltzmann's constant $k = 8.625 \cdot 10^{-5}$ eV, we finally obtain

$$E_g = b \cdot 2k = (0.70 \pm 0.01)\ \text{eV}. \text{(Literature value 0.67 eV)}$$

Fig.3: Conductivity of a semi-conductor as a function of the reciprocal of the temperature

Fig.2: Typical measurement of the probe-voltage as a function of the temperature
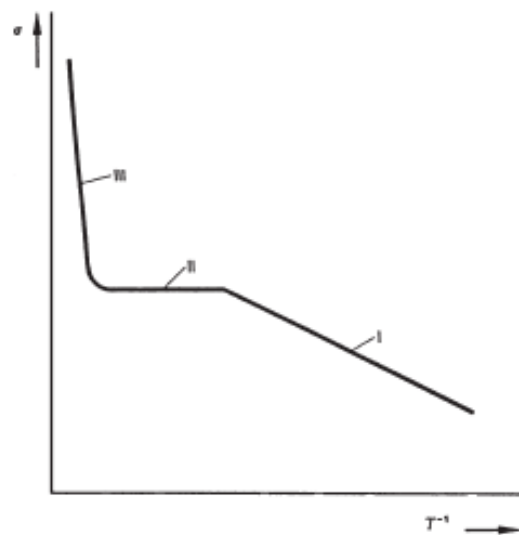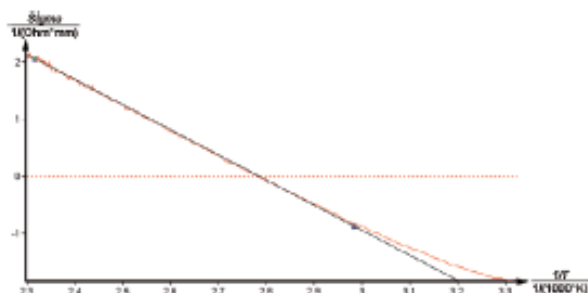

Fig.4: Regression of the conductivity versus the reciprocal of the absolute temperature
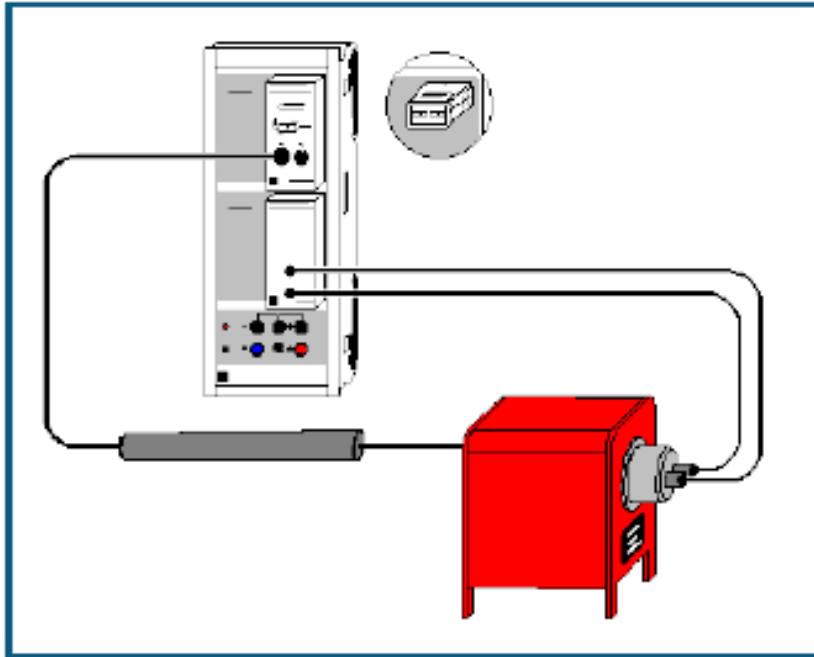
Solid-state physics

Conduction phenomena
Electrical conductivity in solids

Measuring the temperature-dependency of a noble-metal resistor

Description from CASSY Lab 2

For loading examples and settings, please use the CASSY Lab 2 help.

Electrical conduction in solid bodies

 can also be carried out with Pocket-CASSY and Mobile-CASSY

Experiment description

The temperature-dependency of the specific resistance R is a simple test for models of electric conductivity in con-ductors and semiconductors. In electrical conductors, R rises with the temperature, as the collisions of the quasi-free electrons from the conduction band with the incomplete atoms of the conductor play an increasing role. In semicon-ductors, on the other hand, the resistance decreases as the temperature increases since more and more electrons move from the valence band to the conduction band, thus contributing to the conductivity.

This experiment measures the resistance values of a noble-metal resistor and a semiconductor resistor as a function of the temperature. For the noble metal resistor, the relationship

$$R = R_0 \cdot (1 + \alpha \cdot \vartheta) \quad (R_0: \text{resistance at } \vartheta = 0 \text{ °C})$$

is verified with sufficient accuracy in the temperature range under study. For the semiconductor resistor, the evalua-tion reveals a dependency with the form

$$R \propto e^{\Delta E/2kT} \quad (k = 1.38 \cdot 10^{-23} \text{ J/K: Boltzmann constant})$$

with the energy band interval ΔE.

Equipment list

1	Sensor-CASSY	524 010 or 524 013
1	CASSY Lab 2	524 220
1	Current source box	524 031
1	Temperature box	524 045
1	Temperature sensor NiCr-Ni	666 193
	or	
1	NiCr-Ni adapter S	524 0673
1	Temperature sensor NiCr-Ni, type K	529 676
1	Noble metal resistor	586 80
1	Semiconductor resistor 5 kΩ	586 821
1	Electric oven, 230 V	555 81
1	Safety connecting box	502 061

2 Connecting leads, 100 cm, black   500 444
1 PC with Windows XP/Vista/7/8

Experiment setup (see drawing)

The temperature box at Sensor-CASSY input A measures the temperature of the sensor in the electric oven. Insert the measuring tip into the hole on the back of the oven so that the tip is in direct proximity to the resistor element. The current supply box at input B registers the electrical resistance.

Carrying out the experiment

☐ Load settings
- Start the measurement with ⏱ (a value pair is recorded for every temperature increase of 5 K).
- Switch on the oven.
- Stop the measurement with ⏱ at the latest when the temperature reaches 470 K (approx. 200 °C).
- Switch off the oven and remove the resistor.
- When the oven has cooled off repeat the measurement with a different resistor.

Evaluation

When a noble metal (platinum) resistor is used, we obtain a linear increase in resistance as the temperature rises. The temperature coefficient α of the resistor can be determined easily by fitting a straight line. In this example we observe in increase in resistance of 0.407 Ω/K and a resistance of 100 Ω at 0 °C, i.e. α = 0.00407 /K. The agrees very well with the literature value α = 0.00392 /K for platinum.

The resistance of the semiconductor resistor does not decrease linearly as the temperature rises. By fitting a Free Fit we can confirm the relationship $R \propto e^{\Delta E/2kT}$. In the example, this gives us $\Delta E/2k$ = 4000 K for the semiconductor used here, i.e. $\Delta E = 11.0 \cdot 10^{-20}$ J = 0.69 eV (1 eV = $1.602 \cdot 10^{-19}$ J).

Remarks

The measurement will work also during the cooling phase. Then, the error caused by the temperature difference between the temperature sensor and the resistor is smaller, since the cooling phase lasts much longer than the warm-up phase.

When using the old semiconductor resistor (586 82), use the **Settings (noble metal resistor)** for the correct resistance range.

Electron spin resonance

Pre Lab Questions:

1- What is the origin of magnetism? What is the difference between paramagnetic, ferromagnetic and diamagnetic substances?

2- What is Zeeman Effect? And explain what does happen when an external magnetic field is applied on a paramagnetic substance?

3- Drive the equation of (Lande factor)?

4- What is the difference between the electron spin resonance (ESR) and nuclear magnetic resonance (NMR)?

5- What is your goal for the experiment and how are you going to achieve it?

The aims of this experiment are:

➢ Study the electron spin resonance (ESR) in a sample of D.P.P.H (Di Phenyl Picryl Hydrazyl).

➢ Determine the g-factor (Landé-factor) of the DPPH (Diphenylpicrylhydrazyl) specimen.

In your REPORT write down everything you used or found for this experiment.

About your device and experimental process please enjoy the experiment sheets!

Finally:

Discus your results!

Electron spin resonance at DPPH

Determining the magnetic field as a function of the resonance frequency

Objects of the experiment

- Determining the resonance magnetic field B_0 as function of the selected frequency v.
- Determining the g-factor of DPPH.
- Determining the line width δB_0 of the resonance signal

Principles

Since its discovery by *E. K. Zavoisky* (1945), electron spin resonance (ESR) has developed into an important method of investigating molecular and crystal structures, chemical reactions and other problems in physics, chemistry, biology and medicine. It is based on the absorption of high-frequency radiation by paramagnetic substances in an external magnetic field in which the spin states of the electrons split.

Electron spin resonance is limited to paramagnetic substances because in these the orbital angular momenta and spins of the electrons are coupled in a way that the total angular momentum is different from zero. Suitable compounds are, e.g., those which contain atoms whose inner shells are not complete (transition metals, rare earths), organic molecules (free radicals) which contain individual unpaired electrons or crystals with lattice vacancies in a paramagnetic state.

The magnetic moment associated with the total angular momentum $\vec{J}$ is

$$\vec{\mu}_J = -g_J \cdot \frac{\mu_B}{\hbar} \cdot \vec{J} \tag{I}.$$

$(\mu_B = \frac{\hbar \cdot e}{2 \cdot m_e}, \hbar = \frac{h}{2\pi}, \mu_B$: Bohr magneton,

h: Planck constant, g_J: Landé splitting factor,
m_e: mass of the electron, e: electronic charge)

In a magnetic field $\vec{B}_0$, the magnetic moment $\vec{\mu}_J$ gets the potential energy

$$E = -\vec{\mu}_J \cdot \vec{B}_0 \tag{II}.$$

E is quantized because the magnetic moment and the total angular momentum can only take discrete orientations relative to the magnetic field. Each orientation of the angular momentum corresponds to a state with a particular potential energy in the magnetic field. The component J_z of the total angular momentum, which is parallel to the magnetic field, is given by

$$J_z = \hbar \cdot m_J \text{ with } m_J = -J, -(J - 1), \dots , J \tag{III},$$

where the angular momentum quantum number is an integer or a half-integer, i.e. the potential energy splits into the discrete *Zeeman* levels

$$E = g_J \cdot \mu_B \cdot B_0 \cdot m_J \text{ with } m_J = -J, -(J - 1), \dots , J \tag{IV}$$

The energy splitting can be measured directly by means of electron spin resonance. For this a high-frequency alternating magnetic field

$$\vec{B}_1 = \vec{B}_{HF} \cdot \sin(2\pi v \cdot t)$$

which is perpendicular to the static magnetic field $\vec{B}_0$ is radiated into the sample. If the energy $h \cdot v$ of the alternating field is equal to the energy difference ΔE between two neighbouring energy levels, i.e., if the conditions

$$\Delta m_J = \pm 1 \tag{V}$$

and

$$h \cdot v = \Delta E = g_J \cdot \mu_B \cdot B_0 \tag{VI}$$

are fulfilled, the alternating field leads to a "flip" of the magnetic moments from one orientation in the magnetic field B_0 into the other one. In other words, transitions between neighbouring levels are induced and a resonance effect is observed which shows up in the absorption of energy from the alternating magnetic field radiated into the sample.
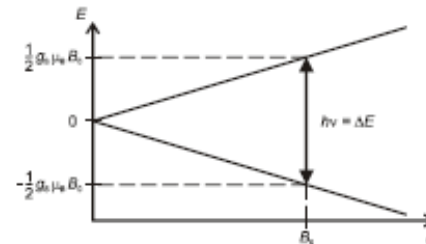

Fig. 1 Energy splitting of a free electron in a magnetic field and resonance condition for electron spin resonance.

In numerous compounds, orbital angular momentum is of little significance, and considerations can be limited to the spin of the electrons. To simplify matters, the situation is represented for a free electron in Fig. 1: here the total angular momentum is just the spin $\bar{s}$ of the electron. The angular momentum quantum number is

$$J = s = \frac{1}{2}$$

and the Landé factor is

$g_J = g_s \approx 2.0023$.

In a magnetic field, the energy of the electron splits into the two levels

$$E = g_s \cdot \mu_B \cdot B_0 \cdot m_s \quad \text{with} \quad m_s = -\frac{1}{2}, \frac{1}{2} \qquad \text{(IVa)},$$

which correspond to an antiparallel and a parallel orientation of the electron spin with respect to the magnetic field. In a transition between the two levels, the selection rule (V) is automatically fulfilled: in analogy to Eq. (VI), the resonance condition reads

$$h \cdot \nu = g_s \cdot \mu_B \cdot B_0 \qquad \text{(VIa)}.$$

If now the energy which is absorbed from the alternating field is measured at a fixed frequency ν as a function of the magnetic field B_0, an absorption line with a half-width δB_0 is obtained. In the simplest case, this line width in a homogeneous magnetic field is an expression of the uncertainty δE of the transition. The uncertainty principle aplies in the form

$$\delta E \cdot T \geq \frac{\hbar}{2} \qquad \text{(VII)},$$

where T is the lifetime of the level. Because of Eq. (V),

$$\delta E = g \cdot \mu_B \cdot \delta B_0 \qquad \text{(VIII)}.$$

Thus the relation

$$\delta B_0 = \frac{\hbar}{2 \cdot g_J \cdot \mu_B \cdot T} \qquad \text{(IX)}.$$

does not depend on the frequency ν. In this experiment, the position and width of the absorption lines in the ESR spectrum of the sample under consideration are evaluated.

From the position, the Landé factor g_J of the sample is determined according to Eq. (VI). In the case of a free atom or ion, the Landé factor lies between $g_J = 1$ if the magnetism is entirely due to orbital angular momentum and $g_J \approx 2.0023$ if only spins contribute to the magnetism. However, in actual fact the paramagnetic centres studied by means of electron spin resonance are not free. As they are inserted into crystal lattices or surrounded by a solvation sheath in a solution, they are subject to strong electric and magnetic fields, which are generated by the surrounding atoms. These fields lead to an energy shift and influence the *Zeeman* splitting of the electrons. Thereby the value of the *g*-factor is changed. It frequently becomes anisotropic, and a fine structure occurs in the ESR spectra. Therefore the *g*-factor allows conclusions to be drawn regarding electron binding and the chemical structure of the sample under consideration.

From the line width, dynamic properties can be inferred. If unresolved fine structures are neglected, the line width is determined by several processes which are opposed to an alignment of the magnetic moments. The interaction between aligned magnetic moments among each other is called spin-spin relaxation, and the interaction between the magnetic moments and fluctuating electric and magnetic fields, which are caused by lattice oscillations in solids and by thermal motion of the atoms in liquids, is called spin-lattice relaxation.

In some cases, the line width is influenced by so-called exchange interaction and is then much smaller than one would expect if there were pure dipole-dipole interaction of the spins.

ESR spectrometers developed for practical applications usually work at frequencies of about 10 GHz (microwaves, X band). Correspondingly, the magnetic fields are of the order of magnitude of 0.1 to 1 T. In this experiment, the magnetic field B_0 is considerably weaker. It is generated by means of the Helmholtz coils and can be adjusted to values between 0 and 4 mT by appropriate choice of the coil current. A current which is modulated with 50 Hz is superimposed on the constant coil current. The magnetic field B, which is correspondingly modulated, is thus composed of an equidirectional field B_0 and a 50-Hz field B_{mod}. The sample is located in an HF coil which is part of a high-duty oscillating circuit. The oscillating circuit is excited by a variable frequency HF oscillator with frequencies between 15 and 130 MHz.

If the resonance condition (V) is fulfilled, the sample absorbs energy and the oscillating circuit is loaded. As a result, the impedance of the oscillating circuit changes and the voltage at the coil decreases. This voltage is converted into the measuring signal by rectification and amplification.

The measuring signal reaches the output of the control unit with a time delay relative to the modulated magnetic field. The time delay can be compensated as a phase shift in the control unit. A two-channel oscilloscope in X-Y operation displays the measuring signal together with a voltage that is proportional to the magnetic field as a resonance signal. The resonance signal is symmetric if the equidirectional field B_0 fulfils the resonance condition and if the phase shift φ between the measuring signal and the modulated magnetic field is compensated (see Fig. 2).
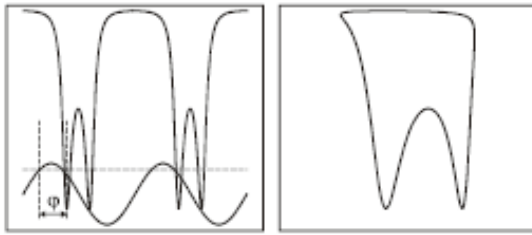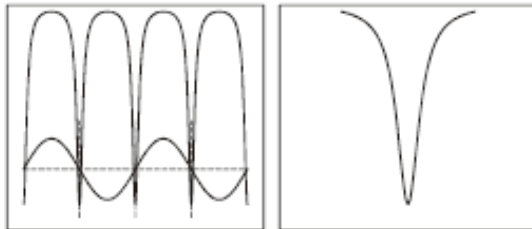
2a
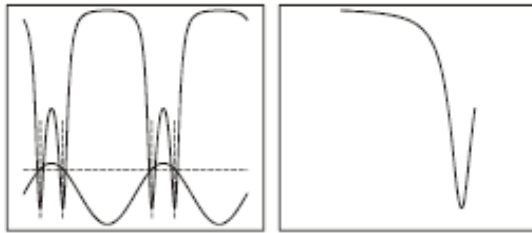
2b
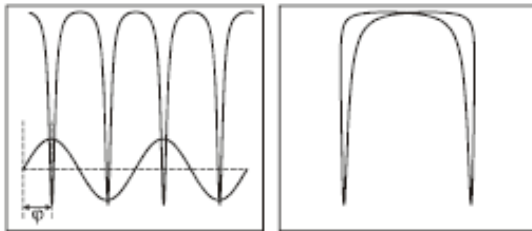
2c

2d

Fig. 2 Oscilloscope display of the measuring signal (Y or I, respectively) and the modulated magnetic field (X or II, respectively)

left: two-channel display with DC coupled channel II
right: XY display with AC coupled channel II

Fig. 2a phase shift φ not compensated, equidirectional field B_0 too weak

Fig. 2b phase shift φ compensated, equidirectional field B_0 too weak

Fig. 2c phase shift φ not compensated, appropriate equidirectional field B_0

Fig. 2d phase shift φ compensated, appropriate equidirectional field B_0

The sample substance used is 1,1-diphenyl-2-picryl-hydrazyl (DPPH). This organic compound is a relatively stable free radical which has an unpaired valence electron at one atom of the nitrogen bridge (see Fig. 3). The orbital motion of the electron is almost cancelled by the molecular structure. Therefore the g-factor of the electron is almost equal to that of a free electron. In its polycrystalline form the substance is very well suited for demonstrating electron spin resonance because it has an intense ESR line, which, due to exchange narrowing, has a small width.
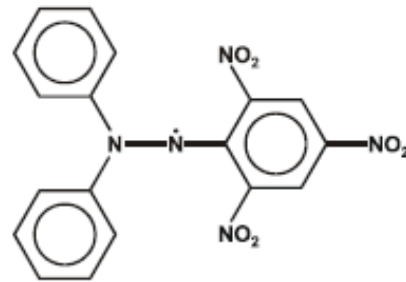

Fig. 3 Chemical structure of 1,1-diphenyl-2-picryl-hydrazyl (DPPH)

Apparatus

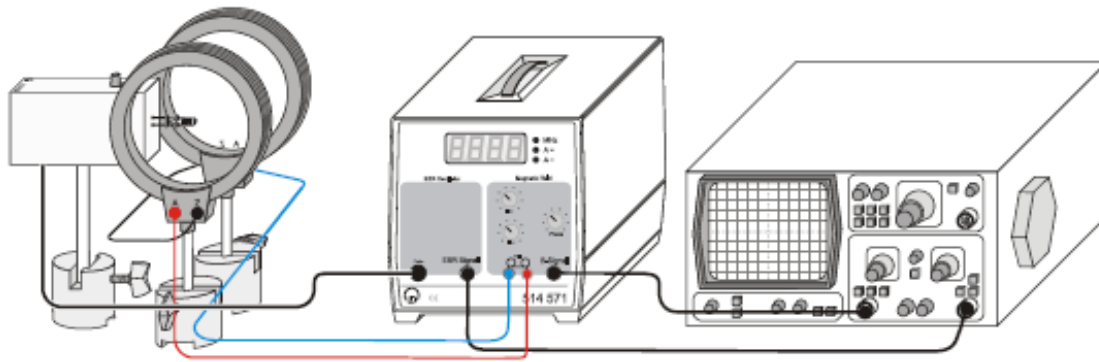|	
1 ESR basic unit	514 55
1 ESR control unit	514 571
1 pair of Helmholtz coils	555 604
1 two-channel oscilloscope 303	575 211
2 BNC cable 1m	501 02
3 saddle bases	300 11
1 connection lead 25 cm black	501 23
1 Connection lead 50 cm red	501 25
1 Connection lead 50 cm blue	501 26

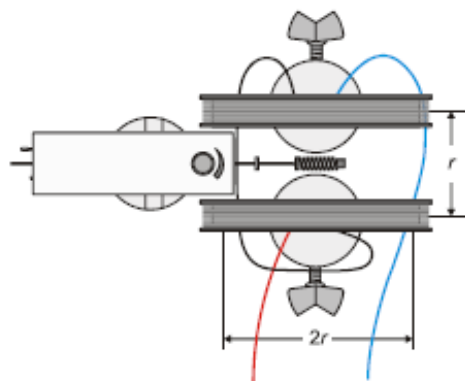Fig. 4 Experimental setup for electron spin resonance at DPPH.

Fig. 5 Arrangement of the Helmholtz coils viewed from above.

Setup

The experimental setup is illustrated in Figs. 4 and 5.

- Set up the Helmholtz coils mechanically parallel to each other at an average distance of 6.8 cm (equal to the average radius r).

- Connect the Helmholtz coils electrically in series to each other and to the ESR control unit, note the details in Fig. 5.

- Connect the ESR basic unit to the ESR control unit via the 6-pole cable.

- Connect the output "ESR Signal" of the ESR control unit to channel II (y-Axis) of the two-channel oscilloscope and the output "B-Signal" to channel I (x-Axis) via BNC cables.

Carrying out the experiment

Determining the resonance magnetic field B_0:

- Put on the plug-in coil 15-30 MHz (the biggest one of the three coils) and insert the DPPH sample so that it is in the centre.

- Switch the ESR basic unit on and set it up so that the plug-in coil with the DPPH sample is located in the centre of the pair of Helmholtz coils (see Fig. 5).

- Set the resonance frequency v = 15 MHz (potentiometer on top of the basic unit).

- Set the modulation amplitude $I\sim$ to the middle.

- Set the phase shift to the right (potentiometer Phase).

- Select two-channel operation at the oscilloscope.

Dual	on	
time base	2	ms/cm
Amplitude I and II	0.5 V/cm AC	

- Use the button on the control unit to switch the display of the control unit to "A=", showing the value of $I=$. Slowly enhance the equidirectional field of the Helmholtz coils with the current $I=$ until the resonance signals are equally spaced (see Fig. 3). At a frequency of 15 MHz this will be at an approximate current of 0.13 A.

- Switch the oscilloscope to XY operation, and set the phase shift so that the two resonance signals coincide (see Fig. 2).

- Vary the direct current $I=$ until the resonance signal is symmetric. Select a modulation current $I\sim$ as small as possible.

- Read the direct current $I=$ through the pair of Helmholtz coils, and take it down together with the resonance frequency v, creating Table 1.

- Increase the resonance frequency v by 5 MHz, and adjust the new resonance condition by increasing the direct current $I=$.

- Again measure the current $I=$ and take it down.

- Continue increasing the high frequency in steps of 5 MHz (use the plug-in coil 30-75 MHz for frequencies greater than 30 MHz and the plug-in coil 75-130 MHz (the smallest one) for frequencies greater than 75 MHz) and repeat the measurements.

Determining the half-width δB_0:

- Select XY operation at the oscilloscope.

Amplitude II	0.5 V/cm AC

- Adjust the resonance condition for v = 50 MHz (medium plug-in coil) once more.

- Extend the resonance signal in the X direction exactly over the total width of the screen (10 cm) by varying the modulation current $I\sim$.

- Switch the control unit display to $I\sim$ and read the RMS (!) value of the modulation current I_{mod}, for example 0.282 A.

- Spread the X deflection(changing to 0.2 V/cm), read the width ΔU of the resonance signal at half the height of the

oscilloscope screen, and take it down, for example 1.5 cm.

Measuring example

Determining the resonance magnetic field B_0

In Table 1, the current through the series-connected Helmholtz coils I_0 in the case of resonance is listed as a function of the frequency ν of the alternating high frequency field.

Table 1: the current I_0 as a function of the frequency ν of the alternating field

$\dfrac{\nu}{\text{MHz}}$	$I = \dfrac{}{\text{A}}$	Plug-in coil
15	0.13	big
20	0.17	big
25	0.21	big
30	0.26	big
30	0.26	medium
35	0.30	medium
40	0.34	medium
45	0.38	medium
50	0.43	medium
55	0.47	medium
60	0.51	medium
65	0.55	medium
70	0.60	medium
75	0.64	medium
75	0.64	small
80	0.68	small
85	0.72	small
90	0.77	small
95	0.81	small
100	0.85	small
105	0.89	small
110	0.94	small
115	0.98	small
120	1.02	small
125	1.06	small
130	1.11	small

Determining the half-width δB_0:

half-width read from the oscilloscope, 1.5 cm corresponding

to : $\delta U = 1.5\,\text{cm} \cdot 0.2\dfrac{\text{V}}{\text{cm}} = 0.3\,\text{V}$

Calibration of the full modulation voltage U_{mod}:

$U_{mod} = 10\,\text{cm} \cdot 0.5\dfrac{\text{V}}{\text{cm}} = 5\,\text{V}$

corresponds to $I_{mod} = 0.28\,\text{A}$ (RMS of AC).

Peak-to-peak Amplitude is $2\sqrt{2}$ of RMS.

Evaluation

The magnetic field B of the Helmholtz coils can be calculated from the current I through each coil:

$B = \mu_0 \cdot \left(\dfrac{4}{5}\right)^{\frac{3}{2}} \cdot \dfrac{n}{r} \cdot I$ with $\mu_0 = 4\pi \cdot 10^{-7}\,\dfrac{\text{Vs}}{\text{Am}}$

(n: number of turns per coil, r: radius of the coils)

With $n = 320$ and $r = 6.8$ cm $B = 4.23\,\text{mT} \cdot \dfrac{I}{\text{A}}$ is obtained.

Determining the resonance magnetic field B_0:

In Table 2 the values calculated for the magnetic field are compiled.

Table 2: The magnetic field B_0 as a function of the frequency ν of the alternating field.

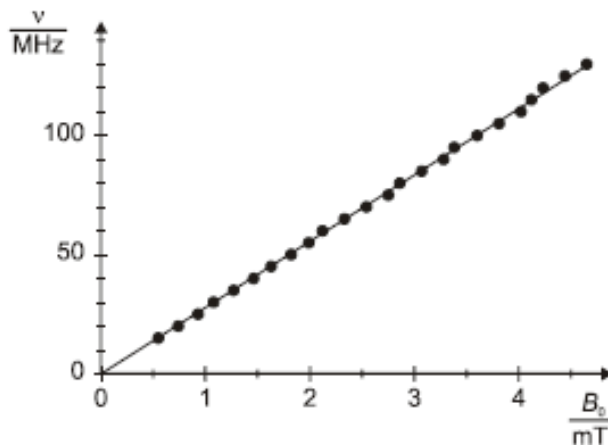| $\dfrac{\nu}{\text{MHz}}$	$\dfrac{B_0}{\text{mT}}$
15	0.55
20	0.74
25	0.93
30	1.08
35	1.27
40	1.46
45	1.63
50	1.82
55	1.99
60	2.12
65	2.33
70	2.54
75	2.75
80	2.86
85	3.07
90	3.28
95	3.38
100	3.60
105	3.81
110	4.02
115	4.12
120	4.23
125	4.44
130	4.65

Fig. 6 the resonance frequency as a function of the magnetic field for DPPH

Fig. 6 shows a plot of the measured values. The slope of the straight line through the origin drawn in the plot is

$$\frac{\nu}{B_0} = 27.8 \frac{\text{MHz}}{\text{mT}}.$$

From this the g-factor follows:

$$g = \frac{h \cdot \nu}{\mu_B \cdot B_0} = \frac{6.625 \cdot 10^{-34} \text{Ws}^2}{9.273 \cdot 10^{-24} \text{Am}^2} \cdot 27.8 \frac{\text{MHz}}{\text{mT}} = 1.99$$

Value quoted in the literature: g(DPPH) = 2.0036.

Determining the half-width δB_0:

$$\delta I = \frac{\delta U}{U_{\text{mod}}} \cdot I_{\text{mod}} = \frac{0.3\,\text{V}}{5\,\text{V}} \cdot 0.28\,\text{A} \cdot 2 \cdot \sqrt{2} = 0.049\,\text{A}$$

From this

$$\delta B_0 = 4.23\,\text{mT} \cdot \frac{\delta I}{\text{A}} = 0.21\,\text{mT}$$

is obtained.

Value quoted in the literature:

δB_0 (DPPH) = 0.15-0.81 mT

The line width strongly depends on the solvent in which the substance has recrystallized. The smallest value quoted in the literature is obtained with CS_2 as solvent.

Report Template for each experiment

Experiment Title

Student Name   Group #

dd/mm/yyyy

1   Objective(s):

Give a brief summary of the purpose of the experiment.

2   Principle(s):

Write briefly the principle of the experiment.

3   Apparatus:

List all the tools and apparatus you used to perform the experiment

4   Data:

In this section, you need to show your experimental results (data tables).

Table 1: Caption is important

5   Graphs:

Here you should include all the graphs you plotted from your data and write a caption for each one.

6   Data Analysis:

In this section, you need to explain the results you obtained in the data section, comment on the behavior of the data, and if there is any anomalies results, try to explain them. Also, explain any calculations you performed in the tables.

7   Calculations:

In this section, you should illustrate your calculations and explain them briefly, Also you may need to include the calculation of the error percentage if required.

8   Conclusion:

Summarize your results and comment on them.