

Faculty of Chemistry and Chemical Engineering

MATERIALS

Laboratory Exercises Manual

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Maribor, 2019

CONTENTS

1. exercise – IDENTIFICATION OF POLYMERS	1
2. exercise – PROPERTIES OF POLYMERS	5
a) SEPARATION OF MIXTURE OF POLYMERS BY COLUMN CHROMATOGRAPHY	5
b) DETERMINATION OF LOW DENSITY POLYETHYLENE DEGRADATION RATE	5
3. exercise – PREPARATION OF GLASS WITH LOW MELTING POINT	8
4. exercise – HYDROLYSIS TEST OF GLASS STONES	10
5. exercise - METALLOGRAPHIC ANALYSIS OF METALS	12
a) STEEL IDENTIFICATION	12
b) GRAIN SIZE DETERMINATION	14
6. exercise – TESTING THE MECHANICAL PROPERTIES OF MATERIALS	22

1. exercise – IDENTIFICATION OF POLYMERS

PURPOSE OF THE EXERCISE

Identification of polymers for plastic processing is very important. It is necessary to know the type of polymer or polymer mixture from which a commercial product is made. During the exercise, some standard tests will be conducted to identify the unknown samples of plastics:

- 1. Test of heating and burning.
- 2. Beilstein test for detecting the presence of chlorine.
- 3. Test for solubility in organic solvents.
- 4. Collection of infrared spectra.
- 5. Final identification based on tests 1 to 4.

EQUIPMENT AND CHEMICALS

- tubes,
- solvents,
- tube holder,
- copper wire,
- litmus paper,
- spoon.

EXPERIMENTAL PROCEDURE

1. Test of heating and burning

Tests must be carried out carefully and always with a small amount of the sample. Some polymers burn very violently, often with an explosion, while others release gas during the burning that should not be inhaled. Therefore, the tests are performed only in a fume hood. The test is performed by careful heating of a clean spoon with one grain of polymer until it starts to emit smoke. The sample is removed from the flame and with moistened litmus paper, the presence of acid or alkali is determined. Also pay attention to the development of the characteristic odour. All observed changes are recorded.

Then place the sample in a warmer area of the flame and observe whether the material is burning, the manner of burning, flame colour and the appearance of the residue. Check if the material after removal from the flame still burns.

The findings must be compared with the data presented in Table 1.1, and afterwards the decision about the type of polymer should be made.

2. Beilstein test for detecting the presence of chlorine

With this method, we can easily determine the presence of chlorine. Pure copper wire with a handle is heated in a colourless flame to a red sheen. Briefly touch the sample that is to be analysed with a hot wire and then place it back over the flame. The green colour of the flame indicates the presence of chlorine or bromine.

3. Test for solubility in organic solvents

To determine the solubility of the polymer, one grain of the plastic material is placed in a test tube, and about 1 cm of solvent is added. Solubility is first tested at room temperature. As a first solvent, we use chloroform. If you do not detect any signs of solubility, continue with dissolution in a water bath (at boiling point). For stirring, use a glass stick. When you find a solvent in which the sample dissolves, add three more grains of polymer. When all the grains are dissolved collect the IR spectrum.

4. Collection of infrared spectra

Prepare the sample that has been dissolved in the given solvent for processing by the IR spectrophotometer. Put the sample on a NaCl plate (care must be taken that the plates do not come into contact with moisture, because these plates are water-soluble). Evaporate the remaining solvent on the plate with a hair dryer until a thin film of polymer remains on the plate. Collect the IR spectrum. According to the presence of characteristic functional groups, suggest the type of polymer. Finally, identify the sample with the "fingerprint" method (all of the sample peaks must exactly match the supplied standard).

5. Final identification

Based on these preliminary tests, identify the unknown polymer.

Table 1.1 shows the properties of polymers that burn until we have outer flame, and those that continue to burn after removing the flame.

Material burns, but goes out after removal of the flame				
polymer	flame colour	smell	other properties	
Melamine-formaldehyde	light yellow with blue- green rim	formaldehyde and fish	hard to ignite, alkaline vapor	
Nylon	blue with yellow top	burnt vegetables	transparent thready melt	
Phenol-formaldehyde (Bakelite)	yellow	phenol, formaldehyde	hard to ignite	
Polytetrafluoroethylenes	yellow	odourless	hard to burn, slowly turns to charcoal, acid vapor	
Polyvinylchloride	light yellow with light green rim	phenol, formaldehyde	hard to ignite, alkaline vapor	
Material	burns and continues to	burn after removal of	the flame	
polymer	flame colour	smell	other properties	
Nitrile rubber	yellow, sooty flame	unpleasant,	/	
Polybutadiene rubber	yellow with blue base, sooty flame	unpleasant,	/	
Polyurethane	yellow with blue base	acidic	/	
Styrene-butadiene (SBR rubber)	yellow, very sooty flame	styrene	1	
ABS	yellow with blue base	styrene	/	
Cellulosic acetate	yellow	acetic acid	acid vapor	
Polyacrylonitrile	yellow	first cyanide-like, then like burning wood	1	
Polycarbonate	yellow, sooty flame	phenol	/	
Polyethylene-terephthalate	yellow, sooty flame	sweet	transparent thready melt	
Polyacetals	light blue	formaldehyde	/	
Polymethyl-methacrylate	yellow with blue base	methyl methacrylate	/	
Propylene	yellow with blue base	candle wax	transparent melt	
Polystyrene	yellow with blue base, very sooty flame	styrene	1	
Polyvinyl-acetate	yellow, sooty flame	vinyl acetate	black residue	
Polyvinyl-alcohol	yellow, sooty flame	unpleasant, sweet	black residue	
Silicon	light yellow-white	odourless	white vapour and white residue	

Table 1.1: Properties of some polymers.

Table 1.2 shows the basic properties of some important thermoplastics.

		POLYETHYLENE			
		low donsity	medium	high	
		low density	density	density	EVA
	extrusion temperature / °C	150-260	150-260	150-315	120-205
properties	extrusion pressure / kp/cm ²	560-2100	560-2100	700-1400	560-1400
important for	compression rate	1.8-3.6	1.8-2.2	2.0	/
processing	linear shrinkage / cm/cm	0.015-0.050	0.015-0.050	0.02-0.05	0.007-0.011
	density / g/cm ³	0.910-0.925	0.926-0.940	0.941-0.965	0.935-0.950
	elongation impact / kp/cm ²	40-160	85-250	215-285	100-200
	elongation at break / %	90-800	50-600	20-1000	750-900
mochanical	elastic modulus / kp/cm	985-2500	1750-3700	4200-12600	140-840
properties	impact resistance (IZOD) / kg cm/cm	doesn't break	27-86	27-106	doesn't break
	hardness, Rockwell	D 41-D 46 (Shore) R 10	D 50-D 60 (Shore) R 15	D 60-D 70 (Shore)	(Shore)
	specific heat / cal/°C/g	0.55	0.55	0.55	0.55
thermal properties	resistance to constant heating / °C	82-100	105-120	120	/
	bending temperature under load / °C at 18.5 kp/cm ²	32-40	40-50	43-55	35
	bending temperature under load / °C at 4.5 kp/cm ²	38-50	50-75	60-88	60-65
electric properties	dielectric resistance, sample 3 mm / volt/0.0025	450-1000	450-1000	450-1000	620-780
	dielelectric constant, 60 cycles	2.25-2.35	2.25-2.35	2.30-2.35	2.50-3.16
	loss factor, 60 cycles	0.0005	0.0005	0.0005	0.003-0.020

Table 1.2: Basic properties of selected thermoplastics.

2. exercise – PROPERTIES OF POLYMERS

a) SEPARATION OF MIXTURE OF POLYMERS BY COLUMN CHROMATOGRAPHY

PURPOSE OF THE EXERCISE

This method is called elution chromatography analysis. The mixture of polymers which have to be separated is applied to o the stationary phase in the column. This mixture is separated with different solvents, which represent the mobile phase. Due to the different adsorption capacity of components or different distribution between stationary and mobile phase, the components of the mixture move through the column with different speed consequently and limited zones are shaped. If the mobile phase flows continuously, the components will be separately eluted from the column. Individual zones in the column are usually well limited and components in the effluent well separated.

ACCESSORIES AND CHEMICALS

- column,
- tubes,
- silica gel,
- solvents,
- glass rod,
- wooden tube holder,
- water bath.

EXPERIMENTAL PROCEDURE

Polymer mixture will be separated by column chromatography. Stationary phase is prepared by mixing 4 tablespoons of silica gel Kieselgel 60 (0,063 - 0.1 mm) with chloroform. The stationary phase is filled into the column. On the top of the stationary phase the mixture of polymers applied and slowly (drop by drop) eluted with different solvents according to increasing polarity. In your case use only two solvents (chloroform and tetrahydrofuran) which dissolve polymers in the mixture.

The eluate is collected in tubes. The water bath is used to evaporate the solvent to the viscous residue. Put the concentrated sample on NaCl plate and completely evaporate the solvent. Collect the IR spectrum and determine the type of polymer.

b) DETERMINATION OF LOW DENSITY POLYETHYLENE DEGRADATION RATE

PURPOSE OF THE EXERCISE

Low Density Polyethylene (LDPE) is produced by high-pressure process at (1500 – 3000) bar. This polyethylene has branched structure. Higher degree of branching of polyethylene reduces the ability of polymer chains to come closer together and to be arranged, consequently the conditions for the formation of crystals get worse. This polyethylene has a low density of (0.91 – 0.925) g/cm³, low melting point or softening point and higher permeability of gases and vapors in comparison to high density polyethylene, which has a lower degree of branching.

External influences (light, heat, mechanical stress, various chemicals, radiation, oxygen, ozone) can affect the polymer degradation Oxidation of polyethylene appears mostly on places where polymer molecule is branched the most. The reaction is autocatalytic.

Thermooxidation of polyethylene leads to the formation of peroxide radicals, from which after the cleavage polyethylene hydrocarbon radicals arise. They react with oxygen to give the peroxide radicals again, which are the basis for the formation of new oxidation products (ketones, aldehydes, esters).

Thus, in the pursuit of carbonyl content by IR spectrophotometry we can monitor the progress of thermooxidation or photooxidation:

start: $RH \rightarrow R' + H'$ $R' + O_2 \rightarrow ROO'$

continuation: $ROO' + R'H \rightarrow ROOH + R'$ etc.

In the absence of light the reaction is extremely slow. This reaction is accelerated under the influence of oxygen, UV light, high-energy radiation, etc.

If we find that polyethylene contains carbonyl at $\lambda = (1715 - 1720) \text{ cm}^{-1}$, trans-vinylene at $\lambda = 966 \text{ cm}^{-1}$ or the vinyl group at $\lambda = (992 \text{ and } 909) \text{ cm}^{-1}$ which indicates that the polyethylene is degraded.

EXPERIMENTAL PROCEDURE

Collect the IR spectrum of an unknown sample (foil) and based on the spectrum, determine whether the sample is degraded or undegraded.

We have recorded infrared spectra of undegraded and four degraded samples of LDPE. All samples are of uniform thickness, d = 0.045 mm. Determine which of the degraded samples LDPE is the most degraded.

- 1. LDPE undegraded
- 2. LDPE degraded thermal + photo 25 h
- 3. LDPE degraded thermal 100 h
- 4. LDPE degraded thermal 300 h
- 5. LDPE degraded UV light Xenotest 246 h

For all degraded samples, calculate the absorption coefficient, *a*, using the formula:

$$a = \frac{\log\left(\frac{\boldsymbol{\varPhi}_0}{\boldsymbol{\varPhi}_t}\right)}{d}$$

where:

d = thickness of the sample (mm), D_0 = flow of incoming light (W) and D_t = flow of transmitted light (W). 2.1

The cleavage rate of polymer chains (degradation) can be monitored by determining the content of olefinic groups:

- trans-vinylene group RCH=CHR absorbs at 966 cm⁻¹ and

- vinyl group RHC=CH₂ absorbs at 992 cm⁻¹ and 909 cm⁻¹.

The mole fraction of trans-vinylene groups is determined by measuring the absorption coefficient of the degraded sample at 966 cm⁻¹, and the fraction of trans-vinylene groups per 1000 C atoms is then determined according to the equation:

$$x(\%_{0}) = a_{(966 \text{cm}^{-1})} \cdot 0,18$$
 2.2

In a similar way, by measuring the absorption coefficient of the degraded sample at 909 cm⁻¹, the mole fraction of vinyl groups can be determined:

$$x(\infty) = a_{(909 \text{cm}^{-1})} \cdot 0,13$$
 2.3

The data (absorption coefficients and mole fractions) for all samples should be collected in a table. Determine the most degraded sample.

3. exercise – PREPARATION OF GLASS WITH LOW MELTING POINT

PURPOSE OF THE EXERCISE

For the preparation of ordinary industrial glass, temperatures over 1400 °C are required. If we select the appropriate components and the corresponding procedure, we can make glass with a lower melting point, $T \approx 800$ °C, on the laboratory scale.

EQUIPMENT AND CHEMICALS

- gloves,
- glasses,
- annealing pot,
- pliers,
- steel plate,
- tripod,
- beaker,
- H₃BO₃,
- Li₂CO₃,
- Na₂CO₃,
- CaCO₃,
- SiO2,
- CuO,
- talc.

EXPERIMENTAL PROCEDURE

The glass will be prepared at a very high temperature, so you will need to work carefully.

The annealing furnace is turned on and set to a temperature of 850 °C. Weight the following chemicals:

H ₃ BO ₃
Li ₂ CO ₃
Na_2CO_3
CaCO ₃
SiO ₂

If you want to colour the glass, add a knife tip of CuO (or some other chemical). To prepare white frosted glass, replace the CaCO₃ in the original recipe with 5 g Ca(PO₃)₂ and reduce the amount of Na₂CO₃ to 0.81 g.

The ingredients in the beaker should be well mixed. Preheat the empty annealing pot for 10 min in the annealing furnace. Add 1 spoon of the mixture to the pot and return it to the furnace for 10 min. Then add another three spoons of the mixture and reheat for 10 min. Once the

mixture melts, slowly add the rest of the mixture. The steel plate, which serves as a mold, must be dusted with talc, so that the glass does not stick to the mold, and then placed together with the full pot into the furnace. After 30 min, put a steel plate with a pair of pliers on a tripod. Quickly pour the molten glass from the pot into the heated mold. After 1 h, remove the glass from the mold. Stress in the prepared glass is eliminated by further heat treatment. Place the glass in an oven at 450 °C. Then the furnace is turned off and the glass is allowed to slowly cool overnight.

4. exercise – HYDROLYSIS TEST OF GLASS STONES

PURPOSE OF THE EXERCISE

This is a test with hot water to determine the chemical stability of glass that comes into contact with aqueous solutions. A certain amount of crushed glass is boiled in distilled water. After cooling, the amount of extracted OH⁻ and Na⁺ ions that are present in the solution (because alkali and alkaline-earth oxides in the glass have been dissolved) are determined. Based on these data, the glass is arranged in the appropriate hydrolysis class.

EQUIPMENT AND CHEMICALS

- mortar,
- two sieves (315 μm in 500 $\mu m),$
- filter paper,
- volumetric flasks,
- Erlenmeyer flask,
- graduated pipettes,
- burette,
- water bath,
- HCl (c = 0.01 mol/L),
- NaCl (c = 0.0254 g/L),
- methyl red indicator.

EXPERIMENTAL PROCEDURE

A glass sample is crushed in a mortar (it is mandatory to use protective glasses). Sieve the glass stones through the sieves (above 500 μ m, below 315 μ m) to obtain 2 g of the material, which remains between the two sieves.

1. Determination of OH⁻ ions and hydrolysis class

Prepare a boiling water bath. Place 2 g of the glass stones in a 50 mL flask and wash several times with 30 mL of water (shake the flask, and carefully decant the water with its residue of fine glass powder). After the last rinse with water, the flask is filled to the neck with distilled water. Hang the flask halfway up the neck in a boiling water bath and start timing. After (3-5) min, the water in the flask reaches 93 °C. After that, close the flask and leave closed for 60 min in the water bath. The flask is first cooled in air and then under running water. Fill it to the mark with distilled water, mix well and pipette into an Erlenmeyer flask 25 mL of that water. Add 2 drops of the indicator (methyl red) and titrate with a solution of HCl (c = 0.01 mol/L) until the colour changes. Consumption of acid is different for various types of glass; the amounts are between V = (0.2 to 1) mL per 1 g of the glass stones. Based on the consumption of acid during titration, the glass can be classified into the appropriate hydrolysis class (Table 4.1), and the quantity of discarded OH⁻ ions per gram of glass stones can be calculated.

Table 4.1: Hydrolysis classes.

Hydrolysis class	Consumption of HCl (c = 0,01 mol/L) / mL
1	0-0.1
2	0.1 - 0.2
3	0.2 - 0.85
4	0.85 – 2.0
5	2.0 - 3.5

2. Determination of the alkali metal ions

The amount of Na⁺ ions is determined by flame spectrometry and expressed, as is customary in the analysis of silicates, by mass concentration of Na₂O in mg/L solution or by mass fraction with unit μ g/g (to the g of glass stones). The measurement is performed at a wavelength of 588.5 nm.

For analysis, use the remainder of the extract after heating in a water bath, which is filtered to a beaker. Pipette 5 mL of this solution to the 50 mL volumetric flask and fill to the mark with distilled water. The solutions for the calibration curve can be prepared from a standard solution of NaCl having a concentration of 0.02542 g/L. Using a burette, dispense in a 100 mL volumetric flask the following quantities of the standard solution and dilute to the mark:

mL NaCl		0.269	mg/L Na₂O
mL NaCl		0.539	mg/L Na₂O
mL NaCl	corresponds to:	0.809	mg/L Na₂O
mL NaCl		1.079	mg/L Na₂O
mL NaCl		1.348	mg/L Na₂O
	mL NaCl mL NaCl mL NaCl mL NaCl mL NaCl	mL NaCl mL NaCl mL NaCl corresponds to: mL NaCl mL NaCl	mL NaCl 0.269 mL NaCl 0.539 mL NaCl corresponds to: 0.809 mL NaCl 1.079 mL NaCl 1.348

Express the results as the quantity of discarded Na₂O per gram of glass stones.

5. exercise - METALLOGRAPHIC ANALYSIS OF METALS

a) STEEL IDENTIFICATION

PURPOSE OF THE EXERCISE

The purpose of metallographic analysis of metals is to determine the structural characteristics of metals and alloys, which have an influence on their quality and usefulness. Among these features are the following:

- type of structure, type and classification of structural components, grain size and secretions,
- the proportion of type, size, and classification of non-metallic inclusions,
- cracks, pores, voids and other defects,
- leaching of alloys and uneven distribution of alloying elements and impurities in alloys.

Metallographic analyses are macroscopic and microscopic. In the former case, we observe the metal with eyes or with a magnifier at magnifications up to about 20 x. For enlargements up to 2000 x, we use an optical microscope, and for magnificatios of 100000 x, an electron microscope.

Samples of steel will be observed with an optical microscope. For microscopic observation, the metal surface must be carefully ground, polished and etched, otherwise observation of the metal is practically impossible.

Microstructure

A prerequisite for successful microscopy is a quality specimen. The main requirements for the observed area are:

- an absence of rips and scratches,
- an uncorrugated surface,
- an absence of terrain between the soft and hard phases.

Fine grinding

The purpose of fine grinding is to remove the damaged layers that occurred during cutting and roughing of the sample. After removing the damaged layer of the sample, a new layer of damage is created, but it is not as deep as the first one. This new layer is removed using finer grinding paper. Grind the sample in stages with increasingly fine grinding paper until you are confident that the remaining damage to the sample will be removed during rough polishing. The grinding papers used in our work are 400, 600, 800, 1000, 1500 and 2000.

Polishing

Figure 5.1 shows the state after the fine grinding. The resulting injuries are lower than before grinding. If polishing is carried out correctly, we get to the line A, which represents the real microstructure.



Figure 5.1: Condition of the sample after fine grinding.

EQUIPMENT AND CHEMICALS

- grinding paper,
- nital,
- tweezers,
- alumina,
- fine cloth.

EXPERIMENTAL PROCEDURE

Grind the surface of the sample using a series of grinding papers. In industry, automatic rotation or vibration grinding is used, which gives a higher quality surface. Grind under running water. Used grinding papers designed for fine grinding of metal or wood. Grinding under running water has the advantage that the water washes away the hard particles torn from paper or samples. This decreases the chance of formation of scratches on the surface. Dry grinding (to prevent the removal of dust from the paper, the paper is coated with paraffin) is used where water corrodes the polished surface or reacts with the individual structural components in the metal. Grinding on dry papers is used in the preparation of soft metal samples, for example, aluminium, lead and their alloys. Samples with hard surfaces (e.g. steel) acquire a fine surface if they are machine polished with alumina No. 1 at a speed of (100 – 300) min⁻¹ (speed of rotation of the polishing disc). Very soft metals can only be metallographically polished by hand and without pressure. Polishing generates microrelief on the surface of the sample, blurs the boundaries between different components of the microstructure and rips out individual phases (e.g. non-metallic inclusions), and therefore should be as short as possible. After polishing, thoroughly wash the sample and dry it.

The final stage in the preparation of the sample is etching. Etch the sample for a few minutes in a solution of nital (3% solution of HNO₃ in ethanol). After etching, rinse the sample with water and dry it well.

Microscopy

The microscope consists of a lens tube, eyepieces, a tripod with microscopic stage and lighting. The microscope is used for observing the surface of metal in reflected light. Before working, adjust the diopter adjustment ring and set the magnification. Place the sample on the microscope stage and sharpen the image. Compare the image with the photos, and the steel is identified in this way.

b) GRAIN SIZE DETERMINATION

PURPOSE OF THE EXERCISE

In special cases, the material consists only of a single crystal, but usually it consists of a large number of crystals. Within the crystal grain, the atoms are arranged in a crystal lattice of a certain orientation: certain grains may have the same crystal lattice, but a different orientation. The field of contact between two crystal lattices is called the crystal boundary (Figure 5.2). Atoms in the crystal boundary have fewer neighbours than atoms within the grains. Energy is required for merging the atoms, while at scission the energy is released. Therefore, the atoms on the crystal boundary have more energy than those within the grains – they are more reactive.



Figure 5.2: Crystal grains and grain boundaries.

Figure 5.3 shows the growth of crystal grains. The density of atoms on the crystal boundary is lower than that within the crystal grains, so alloying elements or other atoms can easily diffuse through the grain boundaries.



Figure 5.3: The growth of crystal grains.

The size and shape of the crystal grains affect the mechanical properties of the material. The impact can be traced, as long as we know the parameters for the determination of grain size. The diameter of the grains is useful information, but it is difficult to determine by microscopic observation. We use the ASTM standard or GS number (Grain size number) for comparison with observation under a microscope at 100 x magnification (Table 5.1).

Crystal grain size GS	Number of grains/in ² at 100 x magnification (mean value)	Number of grains/in ² at 100 x magnification (area)
1	1	_
2	2	1.5 – 3
3	4	3 – 6
4	8	6 – 12
5	16	12 – 24
6	32	24 – 48
7	64	48 – 96
8	128	96 – 192

Table 5.1: Grain size distribution.

There is a relationship:

$$N = 2^{n-1}$$
 4.1

where N is the number of grains per in² at 100 x magnification, and n is the GS number.

Another important piece of information for the material is the surface density of crystal boundaries:

$$\frac{A}{V} = \frac{2 \cdot N}{l}$$

where N/l is the length density of the interfaces between crystal boundaries and the line that you draw on the microscopic image.

In table 5.2 the main parameters are presented, depending on the crystal grain size in the real state.

Crystal grain size	Number of	Number of	Number of grains/10cm ²
GS	grains/mm ²	grains/mm ³	at 100 x magnification
0	8	16	0.56 – 1.2
1	16	45	1-2.4
2	32	128	2 – 5
3	64	360	4 - 10
4	128	1020	8 – 20
5	256	2900	16 - 40
6	512	8200	32 - 80
7	1024	23000	64 - 160
8	2048	65000	128 – 320
9	4096	185000	156 – 640
10	8200	520000	512 – 1280

Table 5.2: The parameters depending on the crystal grain size.

EXPERIMENTAL PROCEDURE

To determine the grain size, we must know the area of the visual field of the microscope at a certain magnification. For this purpose, we will first calibrate the microscope. Insert the eyepiece with a special glass that has a black scale (eyepiece scale). Place the glass with the yellow scale under the microscope. Look through the microscope, and following instructions from the assistant, determine parameters I_{ob} and I_{ok} , which are needed to calculate the distance between the two black bars I:

$$I = \frac{I_{\rm ob} \cdot 10\,\mu\rm{m}}{I_{\rm ok}}$$

Since there are 80 black bars, we get the diameter of the visible area by multiplying the distance from bar to bar by 80. From the known diameter, we can calculate the area of the observed field.

A sample of a nodular alloy NL (grey alloy with spherical graphite) with a chemical composition:

C = 3.75 %	S = 0.009 %	Mg = 0.04 %	Mn = 0.37 %
Si = 2.39 %	Cu = 0.05 %	Ni = 0.17 %	

will be observed under a microscope at 500 x magnification. Count the grains within the determined area and the number of grains which intersect the circle. Determine the following: - crystal grain size (table 5.1 and 5.2),

- surface density of the crystal boundaries,

- orientation and shape of crystal grains (based on the accompanying drawings),
- content of C in steel (Materials).



1. Shape of crystal grains

incompletely granular

completely granular



2. The orientation of the crystal grains

oriented

4

3. Size classes of graphite grains at 100 x magnification

2

a) Lamellas















6. exercise – TESTING THE MECHANICAL PROPERTIES OF MATERIALS

PURPOSE OF THE EXERCISE

This test is used to determine the mechanical properties of metallic materials. On various metal samples, a tensile strength test, compression test, shear test, a test for determining Brinell hardness and a deep draw test will be carried out.

EQUIPMENT AND CHEMICALS

- metal samples - tubes,

- Schubler caliper,
- universal machine for testing mechanical properties.

EXPERIMENTAL PROCEDURE

To conduct the experiments, we use a universal machine for testing the mechanical properties of materials and various metal samples - tubes. With the universal machine for testing mechanical properties, we can load material with up to 23 kN.

1. Tensile strength test

With the tensile test of metallic samples, we can determine the limit of elasticity, the limit of plasticity and the tensile strength of the tested material.

Prior to the test, measure the length, l_0 , and the diameter of the sample, d_0 . Clamp the sample into the machine and load it with uniformly increasing force up to breaking.

Record the maximum force F_m , and re-measure the length, l, and diameter of the sample, d.

Calculate the elongation $\mathit{\varepsilon}$, contraction, $\mathit{\Psi}$, and tensile strength, $\mathit{\sigma}_{\mathrm{m}}$:

$$\varepsilon = \frac{l - l_0}{l_0} \tag{6.1}$$

$$\psi = \frac{A_0 - A}{A} \tag{6.2}$$

$$\sigma_{\rm m} = \frac{F_{\rm m}}{A_0}$$
 6.3

2. Compression test

With the compression test of metal samples, we can determine the limit of plasticity and the compression strength of the tested material.

Prior to the test, measure the length, l_0 , and the diameter of the sample, d_0 . Insert the sample into the machine and load it with uniformly increasing force; we usually avoid destroying the material.

Record the maximum force F_m , and re-measure the length, l, and diameter of the sample, d.

Calculate shrinkage, ε , and compression strength, $\sigma_{\rm t}$:

$$\varepsilon = \frac{l - l_0}{l_0}$$

$$\sigma_t = \frac{F_m}{A_0}$$

$$6.5$$

3. Shear stress test

With the shear test of metal samples, we can determine the shear strength of the tested material.

Prior to the test. measure the length, l_0 , and the diameter of the sample, d_0 . Insert the sample into the machine and load it with uniformly increasing force up to breaking. Record the maximum force F_m .

Calculate the shear strength, τ .

$$\tau = \frac{F_{\rm m}}{2 \cdot A_0} \tag{6.6}$$

4. Brinell Hardness test

The test of hardness is carried out in accordance with the DIN EN 10003 standard. A ball made of hardened stainless steel, which has a diameter, d = 10 mm, is impressed into the metal material with a force, F, and with an impression diameter of d = (2.4 - 6 mm). Record the maximum force, F_{m} , and the value of the Brinell hardness, *HBS*.

5. Deep draw test

The Erichsen deep draw test is used for thin materials. Insert the sample into the machine and load it with uniformly increasing force with a metal ball until the first cracks appear. Record the maximum force $F_{\rm m}$.