TOMSK POLYTECHNIC UNIVERSITY

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MATERIALS SCIENCE PRACTICE

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The study aid contains methodical instructions for laboratory works on discipline "Materials Science" and is intended for training students majoring in the direction 150700 "Mechanical engineering" in English. The laboratory practice is directed to intensification of theoretical knowledge in discipline and allows students to get practical skills of laboratory researches.

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Reviewer

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INTRODUCTION

Studying a theoretical material on the discipline "Materials Science" is accompanied by performance of laboratory works cycle. Laboratory works have high informative value; give students the chance of independent training and acquirement of practical skills in the managing modern equipment. Training effect at laboratory researches performing rises considerably.

At Materials Science and Metal Technology department laboratory works at all sections of discipline are carried out. For this purpose there are the specialised audiences equipped with the modern lab ware. Methodical supporting of laboratory works is presented in the form of methodical instructions, accessible in the TPU library and placed on a university site.

Before laboratory work a student should prepare theoretical part of report, using lab instructions.

Each laboratory work is performed as research of any process, group of materials or a method of materials characteristics testing. Students get tasks for performance of work individually or by small groups (3-5 persons). Students work independently under the guidance of the teacher and the educational master. Everyone contributes the share in acquisition of results. Separate works are spent in the form of business game where all group is involved, but everyone has the role.

Upon completion of laboratory work each student should carry out the analysis of the results and draw conclusions. Reports on laboratory works are defended by each student.

Study aid "Materials Science Practice" is intended for training students majoring in the direction 150700 "Mechanical engineering" in English.

LABORATORY WORK № 1

METALS AND ALLOYS HARDNESS TESTING

The purpose of the laboratory work

- 1. To learn to measure hardness of metal samples by various methods.
- 2. To familiarize with conditions of hardness tests application, with preparation of samples for hardness measurement and with the hardness testers.
- 3. To trace the dependence of metals hardness on composition and structure of a material.

The equipment and materials

Brinell, Rockwell and Vickers testers, microhardness tester; samples of various iron-carbon alloys (steels and cast irons); samples made of copper, aluminum and their alloys; samples of the quenched and tempered steels; grinding wheels and an abrasive paper.

The order of performance

- 1. Study theoretical material on the theme of the laboratory work.
- 2. Familiarize with devices and methods of hardness testing in Brinell, Rockwell, Vickers and microhardness tests.
- 3. Measure hardness of the offered samples of metals and alloys on Brinell and Rockwell testers. Fill your data into tables 1 and 2 accordingly.
- 4. Determine approximate value of tensile strength according to measurement of Brinell hardness.
- 5. Analyze the results of measurements and write a report.

Basic principles

Parts of machines, mechanisms and tools should have certain mechanical properties. Mechanical properties of metals characterize resistance of the material to deformation and fracture under the influence of applied forces. Practically all methods of mechanical properties determination are destroying. Special apparatus are necessary for carrying out the tests; testing processes are rather long, being taken into account rather long process of special samples manufacture.

Mechanical and physical properties depend on many factors: structure of a material, a kind of processing (plastic deformation, thermal treatment) and so on. Therefore, it is necessary to control properties, especially mechanical ones, during manufacturing those or other parts. As it was mentioned above, the most of mechanical tests cannot be used at intermediate stages of manufacture owing to duration and dearness of specimens preparation and duration of the testing process

itself. In this case of hardness tests are used.

Hardness has great practical value, as it reflects many service properties of a material, for example, wear resistance, cutting properties, ability to be processed by grinding or cutting, ability to endure local pressure, etc. Besides it is possible to estimate other mechanical properties (for example, tensile strength, etc.) from hardness data. Hence, there is practically confirmed dependence between hardness and other properties of materials.

Hardness tests of materials are widely spread because they do not require special specimens preparation; the technique of tests is rather simple and the test is nondestructive.

The majority of methods of hardness determination use a small indenter, which is forced into the surface of a material to be tested. The indenters are solid bodies (hardened steel balls, a diamond cone or a diamond pyramid). The depth or size of the resulting prints is measured, which in turn is related to a hardness number. Therefore, *hardness is frequently determined as an ability of a material to resist to intrusion of other solid body into its surface*. However, such definition is not common because there are other methods of hardness determination, based not on indentation, but on scratching, swinging of a pendulum, and other principles.

Brinell, Rockwell, Vickers and microhardness tests are the most widely used. All these methods are based on a plastic deformation of a tested material under indentation. The more material resistance to plastic deformation is the smaller the depth of indentation and the higher hardness of the material will be.

Brinell hardness test is used for soft materials: nonferrous metals and their alloys, annealed steel, cast irons (except white one).

Rockwell hardness test is more often used for very hard materials: for hardened steel, tool alloys, ceramics, firm coatings, including layers of sufficient depth on steels and irons, etc. But it is possible to use this method for testing hardness of rather soft materials.

Vickers' method is well suited for measuring the hardness of parts having small thickness or thin surface layers. Rarely this method is applied for hardness measurement of large-sized specimens.

The method of microhardness usually measures hardness within the limits of separate grains or very thin layers. The last two methods are more often used in scientific researches.

The Brinell's method of hardness testing

Brinell hardness test is based on the indentation of a steel ball into a material to be tested and the subsequent measurement of the indentation diameter (fig. 1).

Brinell hardness (*HB*) is defined by the division of the load P applied to the specimen by the area of a print surface F, i.e.

$$HB = \frac{P}{F}.$$
 (1)



Fig. 1

If you calculate a surface of the print having the form of a spherical segment, HB will be defined by the formula

$$HB = \frac{2P}{\pi D \cdot \left(D \cdot \sqrt{D^2 - d^2}\right)},\tag{2}$$

in which P is the applied load in kg; D is the diameter of a ball in mm and d is the diameter of a print in mm.

The size of a ball is chosen depending on thickness of a specimen. Usually, balls of the standard diameters are used: 10 mm, 5 mm or 2.5 mm.

Load applied to a ball is chosen depending on a type of material and should be proportional to the square of the ball diameter. The standard norms accepted for various materials are the following:

for steels and cast iron $P = 30D^2$: for copper and copper alloys $P = 10D^2$; for lead and tin $P = 2.5D^2$.

More particular recommendations concerning the choice of load and time of its application for various materials are given in the standard table in laboratory work on hardness tests.

Besides mentioned above, the load is considered to be chosen correctly if the following correlation is fulfilled

$$0.2D < d < 0.6D.$$
 (3)

The diameter of a print is measured with the special low-power magnifier or a microscope in two mutually perpendicular directions and than the average arithmetic of two measurements is calculated. The magnifier has a scale, the least division of which (or the price of division) is equal to 0.05 mm. In fig. 2 the measurement of the print diameter with the magnifier is shown.

The Brinell hardness number is determined by the formula (2) according to the results of the print diameter measurement. But usually the table for Brinell hardness numbers definition is applied (the table is given in the appendix 1).



Fig. 2

As it was marked above, there is certain dependence between mechanical properties, in particular, tensile strength TS and Brinell hardness *HB*. It can be expressed by the empirical formula:

 $TS = C \cdot HB \cdot 10 \text{ [MPa]},$

where *C* is the coefficient of proportionality.

For steels	C = 0.33 - 0.36;
for aluminum	C = 0.4;
for copper	C = 0.48;
for aluminum alloys	C = 0.37;
for brass, bronze	C = 0.53.

It is necessary to note, that for brittle materials (cast iron, casting aluminum alloys) there is no reliable correlation between hardness and strength. In particular, for calculation of gray cast iron tensile strength the following empirical formula can be used:

$$TS = \frac{10 \cdot (HB - 40)}{6}$$
 [MIIa].

The Rockwell's method of hardness testing

Rockwell hardness test is based on the indentation of a material being tested with a diamond cone having an angle 120° (scales *A* and *C*) or with a steel ball having the diameter of 1.5875 mm (scale *B*), and on the subsequent measurement of penetration depth *h* (fig. 3).



Fig. 3

The Rockwell hardness is expressed by the abstract magnitude depending on the depth of penetration (h), and can be determined by the formula, but it is not necessary, because the hardness is shown on the tester's scale by the arrow.

The Rockwell's method allows testing parts with hardened surface layers and steel parts after quenching. Virtually, this method of hardness determination does not damage a surface of a material.

Scale *C* serves for testing rather hard materials having Brinell hardness from 230 up to 700 kg/mm². The diamond cone is pressed under the load of 150 kg. The interval of hardness measurement by scale *C* is from 22 up to 68 units, hardness number is designated as *HRC*.

Scale A is used for test of very hard and brittle materials or thin surface layers (0.5-1.0 mm). The same diamond cone is pressed under the load of 60 kg. Hardness number is read on scale C, but hardness designation is *HRA*. Hardness measurement interval in this scale is from 70 up to 85 units.

Scale *B* is intended for testing soft materials having Brinell hardness from 60 up to 230 kg/mm². The steel ball with the diameter of 1.5875 mm is pressed under the load of 100 kg. Hardness is measured in limits from 25 up to 100 units of the scale *B* and hardness number is designated as *HRB*.



The Vickers' method of hardness testing

The Vickers' method is based on indentation of a diamond regular tetrahedral pyramid into the surface of specimen under the load P, and the subsequent measurement of a print diagonal d (fig. 4).

Load *P* can vary from 9.8 N (1 kg) up to 980 N (100 kg). If *P* is measured in N, the Vickers hardness is defined as

$$HV = 0.189 \frac{P}{d^2}$$
 [MPa].

If P is measured in kg, the Vickers hardness is defined as

$$HV = 1.854 \frac{P}{d^2} [\text{kg/mm}^2].$$

Fig. 4

The Vickers' method is used for measuring the hardness of parts of small thickness and thin hardened surface layers.

The thinner material is the less load should be. Vickers hardness number HV is determined using special tables according to the size d (diagonal of a print measured in millimeters).

Microhardness method

Determination of microhardness (in microscopically small volumes) is necessary to measure hardness of very thin coatings, or separate structural constituents of alloys (fig. 5).

The device for microhardness determination consists of the mechanism for pressing of a diamond pyramid under loads and metallographic small a microscope. Diamond pyramid is pressed into a tested surface under loads from 0.05 N to 5.00 N. Microhardness number *H* is determined by the same formula, as the Vickers hardness:



Fig. 5

$$H = 0.189 \frac{P}{d^2}$$
, if *P* is measured in N.

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Tasks and methodical instructions

Before starting hardness measurement it is necessary to choose an appropriate technique of hardness determination (Brinell's or Rockwell's method). For this purpose it is necessary to know the treatment which a specimen was supposed to (annealing, quenching or tempering). If there are no data for orientation, it is possible to try to determine hardness with an ordinary file. If the file slides along a metal surface or only insignificantly scratches it, the specimen has high hardness and then it is necessary to apply the Rockwell's method of hardness measurement. If the sample is well cut by a file (the part of metal is easily removed), it has rather low hardness and then it is necessary to apply the Brinell's method of hardness measurement.

Having chosen the test method it is necessary to prepare a sample for test.

For the Brinell test a sample must be prepared, so that its surfaces (tested and basic) should be parallel, have no scale, rust and other roughness, which could influence the result of test. This may be reached by cutting or processing of the surfaces by an emery paper, grinding wheel or a file without heating. The minimal thickness of a sample should be at least 10 times the print depth.

For the Rockwell hardness testing surfaces of sample (tested and basic) are grinded with fine emery paper or with a fine-grained grinding wheel. Processing should not be accompanied by heating of a sample above 150 °C. The basic surface of the sample should provide its steady setting onto a table of the tester.

For the Vickers and microhardness testing the tested surface of samples is necessarily grinded and polished (sometimes for determination of microhardness of separate grains the sample is exposed to etching). The basic surface needs to be smoothed on an emery paper.

At any test method the sample should not be shifted, shaken or deformed; its surface should have no rough scratches and traces of the previous tests.

					Table 1
Material	Loading P , kg	Ball diameter D , mm	Print diameter d , mm	Hardness, <i>HB</i>	Tensile strength TS , MPa
1	2	3	4	5	6

The results of the Brinell hardness measurements are put in the table 1.

Students should analyze the hardness data. Take into consideration how hardness of ferrous metals and alloys (steels and cast iron) differs from hardness of non-ferrous ones (copper, brass, bronze, aluminum, etc.). Besides that, it is necessary to describe the difference in hardness of pure metals and their alloys. The analysis of the results of the test should be given in the conclusions of the report.

All students should get acquainted with the Rockwell's method of hardness determination and test hardness of the given samples. Using the translation table (the appendix 2) compare the value of the Brinell hardness of quenched steels and steel samples which were not subjected to heat treatment. Put the data into the table 2.

Table 2

Material	Loading P , kg	Hardness, <i>HRC</i>	Value of hardness <i>HB</i> (from the translation table)
1	2	3	4

The contents of the report

1) The name of the laboratory work and its purpose,

2) The equipment and the materials used,

3) A brief description of methods of hardness determination and their applications,

4) Tables of experimental data and conclusions on these data should be given in the report.

Print diameter	Brinell hardness number		Print diameter	Brinell h	ardness	number	
d_{10}	under lo	ad <i>P</i> (kg),	, equal to	d_{10}	under loa	d <i>P</i> (kg),	equal to
or 2d5, or 4d2,5	$30 D^2$	$10 D^2$	$2,5 D^2$	or 2d5, or 4d2,5	$30 D^2$	10 D^2	$2,5 D^2$
2,00	955			4,00	229	76,3	19,1
2,05	910			4,05	223	74,3	18,6
2,10	868			4,10	217	72,4	18,1
2,15				4,20	207	68,8	17,2
2,20	764			4,25	201	67,1	16,8
2,25	735			4,30	197	65,5	16,4
2,30	707			4,35	192	63,8	16,0
2,35	682			4,40	187	62,4	15,6
2,40	659			4,45	183	60,9	15,2
2,45	616			4,50	179	59,5	14,9
2,50	597			4,55	174	58,1	14,5
2,55	579			4,60	170	56,8	14,2
2,60	562			4,65	167	55,5	13,9
2,65	531			4,70	163	54,3	13,6
2,70	516			4,75	159	53,0	13,3
2,75	489			4,80	156	51,9	13,0
2,80	477			4,85	152	50,7	12,7
2,85	455			4,90	149	49,6	12,4
2,90	444			4,95	146	48,6	12,2
2,95	429			5,00	143	47,5	11,9
3,00	415		34,6	5,05	140	46,5	11,6
3,05	401		33,4	5,10	137	45,5	11,4
3,10	388	129	32,3	5,15	134	44,6	11,2
3,15	375	125	31,3	5,20	131	43,7	10,9
3,20	363	121	30,3	5,25	128	42,8	10,7
3,25	352	117	29,3	5,30	126	41,9	10,5
3,30	341	114	28,4	5,35	123	41,0	10,3
3,35	331	110	27,6	5,40	121	40,2	10,1
3,40	321	107	26,7	5,45	118	39,4	9,86
3,45	311	104	25,9	5,50	116	38,6	9,66
3,50	302	101	25,2	5,55	114	37,9	9,46
3,55	293	97,7	24,5	5,60	111	37,1	9,27
3,60	285	95,0	23,7	5,65	109	36,4	9,10
3,65	277	92,3	23,1	5,70	107	35,7	8,93
3,70	269	89,7	22,4	5,75	105	35,0	8,76
3,75	262	87,2	21,8	5,80	103	34,3	8,59
3,80	255	84,9	21,2	5,85	101	33,7	8,43
3,85	248	82,6	20,7	5,90	99,2	33,1	8,26
3,90	241	80,4	20,1	5,95	97,3	32,4	8,11
3,95	235	78,3	19,6	6,00	95,5	31,8	7,96

Table of Brinell hardness test

HV	HB	HRC	HRA	HV	HB	HRC	HRA
240	228	20,3	60,7	510	475	49,8	75,7
245	233	21,3	61,2	520	483	50,5	76,1
250	237	22,2	61,6	530	492	51,1	76,4
255	242	23,1	62,0	540	500	51,7	76,7
260	247	24,0	62,4	550	509	52,3	77,0
265	252	24,8	62,7	560	517	53,0	77,4
270	256	25,6	63,1	570	526	53,6	77,8
275	261	26,4	63,5	580	535	54,1	78,0
280	266	27,1	63,8	590	543	54,7	78,4
285	271	27,8	64,2	600	552	55,2	78,6
290	275	28,5	64,5	610	560	55,7	78,9
295	280	29,2	64,8	620	569	56,3	79,2
300	285	29,8	65,2	630	577	56,8	79,5
310	294	31,0	65,8	640	586	57,3	79,8
320	304	32,2	66,4	650		57,8	80,0
330	313	33,3	67,0	660		58,3	80,3
340	323	34,4	67,6	670		58,8	80,6
350	332	35,5	68,1	680		59,2	80,8
360	342	36,6	68,7	690		59,7	81,1
370	351	37,7	69,2	700		60,1	81,3
380	361	38,8	69,8	720		61,0	81,8
390	370	39,8	70,3	740		61,8	82,2
400	380	40,8	70,8	760		62,5	82,6
410	390	41,8	71,4	780		63,3	83,0
420	399	42,7	71,8	800		64,0	83,4
430	408	43,6	72,3	820		64,7	83,8
440	418	44,5	72,8	840		65,3	84,1
450	423	45,3	73,3	860		65,9	84,4
460	432	46,1	73,6	880		66,4	84,7
470	442	46,9	74,1	900		67,0	85,0
480	450	47,7	74,5	920		67,5	85,3
490	456	48,4	74,9	940		68,0	85,6

Table for hardness value comparison

LABORATORY WORK № 2

CRYSTALLIZATION. ITS INFLUENCE ON STRUCTURE AND PROPERTIES OF METAL

The purpose of the laboratory work

- 1. Study the mechanism and kinetics of crystallization process.
- 2. Study macrostructures of metal ingots.
- 3. Study the influence of crystallization conditions on structure and mechanical characteristics of metal.

The equipment and materials

Water solutions of salts (potassium bichromate $K_2Cr_2O_7$, potassium permanganate KMnO₄, sodium chloride NaCl), biological microscopes, metal bullions, an alloy of aluminum with silicon (silumin), a muffle electric furnace with a thermocouple and a self-balancing potentiometer, a metal mould, a ceramic mould, a pendulum hammer MK30.

Basic theoretical principles

Crystallization is a process of transferring of liquid substance into a solid, crystalline state. In melted metal there is no long-distance order: distances between atoms are not constant, volumes with the ordered arrangement of atoms arise and disappear again (fig. 1, *a*). During crystallization *long-distance order* is established: atoms are on fixed distances along each dimensional direction, forming *a crystal lattice* (fig. 1, *b*). Such an ordered arrangement arises due to the equilibrium of attractive and repulsive interatomic forces; it corresponds to a minimum of interaction energy of atoms in metal.



Fig. 1 Arrangement of atoms in liquid (a) and solid (b) metal

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Process of crystallization can develop spontaneously, and may be caused by any artificial reasons.

Thermodynamic processes take place spontaneously, only if they result in decrease of free energy of a system. (*Free energy* F is a changeable part of the total energy of the system.) With an increase of metal temperature its free energy drops both in liquid and solid states, but with different rates: the free energy of melted metal drops faster (fig. 2). Therefore the curves of a free energy against temperature cross at any temperature T_s . At any temperature that is lower than T_s solid metal has a lower level of free energy than liquid one, therefore, under cooling a melted metal down to the temperature $T_1 < T_s$, crystallization will begin. And on the contrary, at temperatures higher than T_s liquid state is equilibrium as melted metal has a lower level of free energy. Therefore, heating up to $T_2 > T_s$ will result in melting.



Fig. 2 The dependences of free energy of metal on the temperature

The temperature T_s at which free energy of metal in solid and liquid state is equal is called *theoretical crystallization temperature*. At this temperature the liquid and the crystals are in equilibrium; neither melting, nor crystallization go up to the finish.

In order to provide crystallization process, some overcooling is necessary relatively to theoretical crystallization temperature. In this case formation of crystals yields a gain in free energy, and that is a stimulus of the process. Therefore, actually crystallization always occurs at the temperature below T_S . The real crystallization temperature T_r is always less than theoretical one. The difference between the theoretical and the real crystallization temperature is called *degree of super cooling* ΔT :

$$\Delta T = T_S - T_r$$

Taking the metal temperature during crystallization, it is possible to receive *the cooling curve* (fig. 3). It is the graph of the temperature against the time. This curve

has the horizontal step corresponding to metal transformation from liquid state into solid. The constant temperature at this step is supported by extraction *of latent heat of crystallization*.



Fig. 3 Cooling curve of molten metal

The degree of super cooling at crystallization is not constant. It grows with the increase in cooling rate of metal. Under shop conditions of castings manufacture the degree of super cooling usually does not exceed 20-30 °C, but it can reach several hundred degrees for very pure metals.

The mechanism of crystallization process can be presented as two elementary processes:

1) Formation of nuclei in a liquid (nuclei means the smallest solid particles – centres of crystallization).

2) Crystal growth from the nuclei arisen.

Rates of these elementary processes depend on the degree of super cooling ΔT or the cooling rate V. This dependence is shown in fig. 4.

As it was already mentioned, in most cases the process of metal crystallization occurs upon rather small degrees of super cooling - for instance, at metallurgical plants and casting shops (the left part of the graph in fig. 4).

The curves in fig. 4 make it visible that upon small cooling rates and small super cooling (for example, ΔT_1 and V_1), a low rate of nucleating is received (*n.f.*₁). Hence, in a liquid a small number of nuclei are formed and a small number of crystals grow from them. This results in coarse-grained structure after a solidification of metal. Schematically the structure is shown in fig. 5.

If crystallization occurs upon big cooling rates and big super cooling $(\Delta T_2, V_2)$, a large amount of nuclei is formed in a liquid $(n.f._2)$; therefore, a large number of crystals grow from them. Metal, thus, becomes fine-grained (fig. 6).



Fig. 4 Dependence of crystallization process rate on degree of super cooling



Fig. 5 Crystallization upon slow cooling: a – the beginning of process; b – the finishing of process



Fig. 6 Crystallization upon rapid cooling: a – the beginning of process; b – the finishing of process

Collision of growing crystals results in the loss of the regular shape by them. Such crystals which have lost the regular shape are called *grains*.

If the cooling rate is very small (the extreme left point in fig. 4) only one nucleus can appear, and a *single crystal* grows. Single crystals of semiconductors necessary in electronics are grown by very slow pulling of a seed from a smelt.

At huge cooling rates (an extreme right point in fig. 4) such a big overcooling is reached, that thermal motion of atoms in metal becomes slower, atoms have no time to form long-distance order and to build up a crystalline lattice. The disordered liquid state of metal appears as though "frozen". Metal becomes solid, but has no crystalline structure. It is *amorphous metal* or *metallic glass*. Amorphous metals are widely applied in the modern audio equipment due to unique magnetic properties.

As the structure of metal determines its properties, the regularities of crystallization can be utilized for forming necessary properties of metal in the course of the casting process.

Fine-grained metal has higher resistance to deformation and greater toughness. The matter is that grain boundaries are barriers for development of deformation and for propagation of a crack. In fine-grained metal the integral surface of grains per a unit volume is more than in coarse-grained one, therefore resistance both to deformation and fracture is larger. It requires expending more energy for the finegrain metal fracture.

Therefore, when casting the parts that experience significant loadings under service, it is reasonable to carry out crystallization process under a high cooling rate in order to obtain fine-grained structure. If it is impossible to provide necessary rate of crystallization (in mould pieces of the big mass), they introduce small amounts of impurities in molten metal for grain refinement. These impurities are non-soluble in a melt and thus increase the number of nuclei. Such process is called *modifying*, and impurities are *modifiers*.



Small particles of carbides, oxides $(Cr_7C_3, VC, TiC, Al_2O_3, etc.)$ are usually used as modifiers. Also some substances facilitating nuclei appearance in liquid (B, Mg, etc.) are applied. Grain refinement is promoted also by the vibration of metal while crystallizing.

Fig. 7 Characteristic zones of the ingot:

1 – ingot skin (a zone of small variously oriented crystals);

2 - a zone of the columnar grains growing in the direction, opposite to the direction of heat removal;

3 – a zone of large, variously oriented grains;

4 – a bubble at the top of an ingot

It is necessary to take into consideration, that under castings manufacture the solidification of metal occurs with different cooling rates in different parts of the mould. Cooling rate diminishes from a wall of the mould to its central part, thus, the direction of heat removal varies in places of crystal growth too. As a result, the ingot has a nonuniform structure in cross-section, so, its properties are different too (fig. 7). The surface layer is fine-grained (i.e., it has high mechanical characteristics), but the

central area is coarse-grained.

The size of these zones varies essentially depending on the composition of a metal and conditions of crystallization.

Tasks and methodical instructions

- 1. Learn the basic data on a lab theme.
- 2. Using a biological microscope watch a crystallization development for different supersaturated water solutions of salts. Draw initial, intermediate and final stages of crystallization; underline the characteristic shape of crystals of each salt. Pay attention to places of a solute drop where crystallization process begins earlier and runs faster. Describe and explain results of your observations.
- 3. Study and draw a macrostructure of the ingot. Point zones of crystallization in a figure and explain their formation.
- 4. Melt an aluminum alloy and pore it into metal and ceramic moulds. After solidification and cooling of ingots, test them by impact using a pendulum hammer. Draw macrostructures of fracture surfaces. Explain the difference in the structures and the properties (impact toughness) of the ingots crystallized in metal and ceramic moulds.

The contents of the report

- 1. The purpose of the laboratory work.
- 2. Basic points on a lab theme (briefly).
- 3. Description of separate stages of work. Necessary explanations, numerical data, and drawings.
- 4. The analysis of the results.
- 5. The conclusion.

Test questions

- 1. What is the moving force of the crystallization process?
- 2. What is the mechanism of crystallization of metal?
- 3. What is the extent of a super cooling?
- 4. Why an ingot structure is not equal on cross-section?
- 5. How does cooling rate influence metal structure upon crystallization?
- 6. How is it possible to receive fine-grained metal?
- 7. How does the metal structure influence mechanical characteristics?
- 8. Why properties of coarse-grained and fine-grained metal are different?

LABORATORY WORK № 3

PLASTIC DEFORMATION, COLD-WORKING AND RECRYSTALLIZATION OF METALS

The purpose of the laboratory work

1. To study the influence of plastic deformation on the structure and mechanical properties of metals.

2. To study the influence of heat on properties of the strained metal.

The equipment and materials

1. Heating furnaces with automatic devices for temperature regulation.

- 2. A pneumatic forging hammer.
- 3. Brinell hardness testers.
- 4. Samples of commercially pure copper.

The order of performance

1. Look through work instructions.

2. Execute the experimental part of the work according to the task.

3. Analyze the results and make conclusions according to the theoretical material.

4. Solve one of the problems (page 20) and explain the solution.

Basic theoretical principles

Plastic deformation of metals

The major and most typical property of metals is *ductility* – the ability to undergo a large strain without failure. This property combined with high strength makes metals irreplaceable materials for modern engineering. If the strain disappears after loading removal, it is an *elastic deformation*, and if it remains, it is a *plastic deformation*.

When metal experiences elastic deformation, the distance between atoms in a crystal lattice varies under the influence of external force. When the applied load is released, the reason of interatomic separation change is removed; atoms go back to their places, and the strain disappears.

The plastic deformation is a much more complicated process which takes place under stress exceeding the elastic limit of metal. Finally, the plastic deformation represents a shear of one part of a crystal in respect to another. What is the mechanism of this phenomenon? It is natural to consider a simultaneous displacement of all atoms in one atomic layer in relation to atoms of the next layer along the shear plane MM (fig. 1). Thus, sheets of paper slide if the upper part of a bundle is sheared. It is possible to count a force which should be applied for realization of such a shear, and thus define theoretical strength. Such calculation has been made by J.I. Frenkel. It showed that iron strength should be equal to 1300 kg/mm², whereas actually tensile strength of iron is 15 kg/mm², i. e. 100 times less.











Fig. 2







a





d

b Fig. 3



С

21

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The explanation of the real mechanism of shear processes is given by the theory of *dislocations*. Dislocations are the linear defects, or imperfections of a crystal lattice. The concept of dislocations has been introduced into a physics of metals in order to explain the disparity between observable strength and theoretical one, and to describe the atomic mechanism of slipping in the process of plastic deformation in crystals. If at the beginning dislocation theory was a hypothesis, then direct demonstrations of their existence were gained, and now there are numerous observed data of dislocations.

The simplest and most evident way of dislocations formation in a crystal is a shear (fig. 2a). If shear has occurred only in a part of slip plane and embraces stage ABCD, then boundary line AB between a section, where slipping has already occurred, and not disturbed section in a slip plane is a dislocation. The atomic plane perpendicular to a slip plane and passing through AB is as though superfluous; it is termed an *extra half-plane*, and dislocation AB is called an *edge dislocation* and is designated by the symbol \perp . Other types of dislocation, for example, *screw* (fig. 3*a*) or *mixed* (fig. 4a) exist, too. The screw dislocation has gained its name because the crystal can be considered consisting of one atomic plane twisted as a helical surface around the dislocation AB (fig. 3a). It is easy to see how a motion of dislocations through a crystal causes residual deformation of a crystal (fig. 2-4, b, c, d). Migration of dislocations occurs according to a scheme represented in fig. 5. It shows that while dislocation moves one atomic distance, each atom in extra half-plane and in planes of a bottom part of the crystal has significantly less displacement. Due to that atoms move in turns for distances less than interatomic ones, the dislocation slips for major distances through the crystal. For simultaneous shear of an upper part of a crystal relatively to lower one, it is necessary to overcome interatomic bonds between all boundary atoms on both sides of a slip plane (fig. 1); motion of dislocation to the neighbouring plane requires breaking interatomic bond between two chains of atoms only (fig. 5). It can explain low experimental value of a breaking off stress in crystals.

It is interesting that in wildlife the dislocation principle of motion is used too; for example, snakes and caterpillars usually creep making a hump ("the plus dislocation") near the tail and propelling it towards the head.

Cold-working and recrystallization of metals

The most impressive property of metals under plastic deformation is the strain hardening, or ability of a metal to become stronger as it is plastically deformed. The dislocation theory shows that for strengthening of a metal it is necessary to reduce the mobility of dislocations. There are some ways of restricting dislocations movement; one of them is a strain. An elementary introduction of a dislocation into a crystal by shear displays that the plastic deformation increases a number of dislocations in a crystal. The stronger the applied force is, the more dislocations are produced. At the initial stage the strain occurs due to slipping a relatively small number of dislocations. In the course of a straining they move through a crystal and can be fixed by various barriers. Such fixed dislocations hinder motion of other dislocations, i.e. the strengthening is created by dislocations themselves. This phenomenon is called *strain hardening* (synonyms are: *cold-working, work hardening*) of a metal. The plastic deformation makes essential impact on mechanical properties of metal and its structure (fig. 6).



Fig. 6 Change of structure and properties of the strained metal depending on strain

Fig. 6 displays how under the influence of the applied loading grains of which all technical metals consist, start to deform and stretch, saving the volume. It is a structurally unstable state. Besides, inside each grain and along its boundaries a great number of dislocations are concentrated. The dislocation density increases from $10^{6}-10^{7}$ sm⁻² (for not strained metal) to $10^{10}-10^{12}$ sm⁻² (for the strained one). That is, the crystal lattice of grains becomes distorted, imperfect. With the growth of percent cold work the strength increases, and the ductility diminishes, that leads, under a major percent cold work, to cracks origin and fracture.

For removal of work hardening the metal being deformed is heated; as a result there are processes of redistribution and reduction of structural imperfections density. These processes are *recovery*, *polygonization* and *recrystallization*. The final and strongly acting process transferring cold-worked metal into a stable state is a recrystallization. *The recrystallization* is a process of the full or partial substitution of the strained grains with others, more perfect grains of the same phase (fig. 7). The recrystallization consists of the formation of a new set of strain-free and equiaxed grains and their growth until they completely replace the parent, less perfect material. The processes involve short-range diffusion.



Fig. 7 Change of structure and properties of the strained metal under heating

The least temperature at which process of recrystallization begins and decrease of strength occurs is called a *recrystallization temperature*. Between the recrystallization temperature (T_r) of a metal and its absolute melting temperature (T_m) there is the simple dependence defined by the metal scientist A.A. Bochvar:

$$T_r = \alpha \cdot T_m$$
 (K

The recrystallization temperature of metals and alloys is given below:

 $T_r = (0.1 - 0.2) \cdot T_m$ – for pure metals,

 $T_r = 0.4 \cdot T_m$ – for commercially pure metals,

 $T_r = (0.5-0.6) \cdot T_m$ – for alloys (solid solutions).

The temperature of recrystallization beginning can be defined by the metallographic analysis and X-ray diffraction methods, and also by the change of

properties. If T_r is defined by the hardness change, then a temperature, at which the increase of hardness created by the strain diminishes twice, is accepted as T_r (fig. 8).





It is necessary to know the recrystallization temperature for determination of a *recrystallization annealing* mode, i.e. heat treatment for removal of work hardening. The temperature of such annealing should exceed the recrystallization temperature for the alloy.

The size of the recrystallized grains after annealing depends strongly on percent cold work (fig. 9).

There is a small degree of deformation (less than 10 %), called the critical degree of cold work, after which the size of new recrystallized grains exceeds one of the initial unstrained grains several tens times. This phenomenon is widely spread in engineering where the strain varies from 0 and to major values; it should be taken into consideration since in some cases the grain growth is undesirable (impact strength decreases dramatically), and in others it is favorable (to obtain necessary electromagnetic properties, hot-resistance raise).



Fig. 9 Percent cold work influence on recrystallized grains size

The recrystallization under the critical degree of cold work is caused by a consolidation of groups of grains into one grain. While the degree of cold work is increasing, the recrystallized grains size diminishes, that is connected with the enlargement of the amount of recrtstallization centres.

Plastic deformation of metals can be classified as cold and hot one, concerning the recrystallization temperature.

Cold deformation is carried out at the temperature below the recrystallization one. Cold deformation enlarges the dislocation density, grains stretch in a stress direction; the strength of metal increases, otherwise ductility decreases.

Hot deformation is carried out at the temperature above the recrystallization one. At hot working by pressure (rolling, forging, die forming, pressing) the hardening created in the deformation process is removed as a result of the recrystallization while being deformed.

Tasks and methodical instructions

(For groups of 2-4 persons)

1. Define the recrystallization temperature of copper according to the change of hardness after heating it up to various temperatures (using graphical dependence "temperature –hardness"). Take the samples of copper 30–40 % cold worked. Accept the recrystallization temperature of copper defined by Bochvar formula as the centre of temperature interval. A temperature step from the centre of the interval towards temperature reduction and increase accept as 50 °C. The melting temperature of copper is 1083 °C. Compare the theoretical recrystallization temperature of copper with the one defined during the process and explain the cause of the disparity.

2. Carry out cold working of samples made of commercially pure copper with various deformation degrees. Define the change of hardness of the strained samples depending on the cold work degree.

Calculate the cold work percent from the formula



in which h_0 is a thickness of the sample before deformation,

 $\varepsilon = (h_0 - h_k) \cdot 100 / h_0, \%,$

 h_k is a thickness of the sample after deformation.

Put the results of measurements into the table 1 and draw the graphic dependence of hardness on the cold work degree.

I auto I

Sample	Initial thickness h 0, mm	Final thickness h _k , mm	Cold work degree <i>ɛ</i> , %	Hardness, <i>HB</i>	Note

3. Carry out hot work of samples made of commercially pure copper with various deformation degrees. Define the hardness change of the strained samples after the hot work depending on a work degree. Put the results of measurements into the table 1 and draw the graphic dependence of hardness on degree of deformation.

4. Perform the recrystallization annealing of copper samples after the cold deformation for 10 minutes. Define the hardness change of annealed samples depending on the deformation degree. Put the results of measurements into the table 1 and draw the graphic dependence of hardness on degree of deformation.

Problem tasks

1) Explain the ancient circus number: why the horseshoe bent by the athlete was offered to be unbent to spectators, not vice versa?

2) Stamped parts made of low-carbon steel received under a cold condition, had after die stamping unequal hardness in various sites: it varied from 120 *HB* to 200 *H*B. Hardness of steel before stamping was 100 *HB*. Explain why steel received unequal hardness?

3) Explain whether it is possible to distinguish the metal deformed under a cold condition and the metal deformed under a hot one examining microstructure. What is the difference in microstructure?

4) Three samples of low-carbon steel were exposed to cold deformation: the first got 5 % cold work, the second -15 % and the third -30 %. Then the samples were heated up to 700 %. What sample had the larger grain size? How does grain growth influence steel properties?

5) Explain why it is not recommended to perform last operation of hot processing by pressure with small reduction degree. How can such deformation influence the grain size and properties of a metal?

6) Explain whether it is possible to create considerable hardening of lead, if subject it to be deformed under room temperature. (t_m of Pb is 327.4 %.)

7) Specify how repeated Brinell test in immediate proximity to the previous site will affect the value of hardness.

8) Drawing of wire is carried out for several steps. If the drawing is made without intermediate annealing operations, the wire at the last steps gives ruptures. Explain the reasons of these ruptures and specify measures for their prevention.

9) A rod of brass bent under cold condition is subjected to recrystallization for work hardening removal. Explain whether the rod will have identical grain size on all section after recrystallization or not?

10) Explain what kind of deformation – cold or hot – is:

rolling of tin at room temperature (t_m of Sn is 232 ?C);

steel deformation at 400 % (t_m of steel is approximately 1500 %).

The contents of the report

1. The name of the work and its purpose.

2. The equipment and the materials used for work performance.

3. Basic concepts of deformation, strain hardening and the recrystallization of metals.

4. The description of an experimental part of the work and the results with necessary graphs, analysis, conclusions.

5. The proved decision of the problem.

LABORATORY WORK № 4

STRUCTURE OF PLAIN CARBON STEELS AND CAST IRONS

The purpose of the laboratory work

- 1. Study the state diagram iron carbon.
- 2. Become familiar with the method of the optical microstructural analysis.
- 3. Study microstructures of annealed carbon steels and cast irons.

The equipment and materials

- 1. Metallographic microscopes.
- 2. Collection of samples of steels and cast irons.
- 3. Album of microphotographs of carbon steels and cast irons.

The order of performance

- 1. Read attentively the basic data on the theme of the laboratory work.
- 2. Understand the state diagram iron carbon.
- 3. Study the photos of microstructures in the album.
- 4. Using microscope, study microstructures of several samples belonging to various classes of steel and cast iron. Determine which of the named classes each sample belongs to. Sketch the scheme of the microstructures of the considered samples and designate structural components by figures.

The contents of the report

- 1 The name and the purpose of the work.
- 2 The diagram iron carbon with the description of phase transformations.
- 3 The brief characteristic of the basic classes of carbon steels and cast irons, and their structures.
- 4 Steels structures schemes and cast irons investigated under a microscope, with the indication of structural components and comments.
- 5 Conclusions.

The basic principles

Iron-carbon diagram

On the iron–carbon state diagram processes of the crystallization of iron–carbon alloys (steels and cast irons) and transformations in their structures are considered at slow cooling to room temperature. The diagram (fig. 1) shows the phase constituents and structure of alloys with the concentration from pure iron up to cementite (6.67 wt% C).



Fig. 1 The iron – carbon diagram: *L* is liquid solution; austenite is solid solution of carbon in γ -Fe; cementite is chemical compound Fe₃C; ferrite is solid solution of carbon in α -Fe; pearlite is eutectoid mixture of ferrite and cementite; ledeburite is eutectic, mixture of austenite and cementite; below *PSK* line ledeburite is a mixture of pearlite and cementite

The crystallization of liquid alloy begins at the temperatures corresponding to a *liquidus* line *ACD*. The *solidus* line *AECF* corresponds to temperatures of the end of crystallization. At the temperatures corresponding to a line *AC*, austenite crystallizes from a liquid, and at the temperatures of line *CD* cementite crystals appear (we usually call them *primary* cementite). In the point *C* at 1147 °C liquid alloy containing 4.3 wt% of carbon forms crystals of austenite and primary cementite simultaneously. A mixture of these two types of crystals being eutectic is called *a ledeburite*. At the temperatures corresponding to the solidus line *AE*, alloys with carbon content of up to 2.14 wt% finally crystallize forming austenite. At the temperatures of line *ECF* (an eutectic transformation line) alloys with carbon content from 2.14 up to 6.67 wt% finally crystallize with the formation of eutectic (ledeburite). The structures formed earlier from a liquid solution, namely austenite in the interval from 4.3 wt% C, and primary cementite in the interval from 4.3 to 6.67 wt% C are also present in microstructure of these alloys.

Under further cooling solid alloys undergo phase transformations at the temperatures corresponding to lines *GSE*, *PSK* and *GPQ*. Transformations in a solid state occur owing to polymorphic transformation γ Fe into α Fe and in connection with downturn of solubility of carbon in austenite and ferrite. With decreasing in temperature excessive carbon is produced in the form of cementite from a solid solution.

Austenite occupies the AGSE field of diagram. Under cooling to the

temperatures corresponding to the line GS, austenite decomposes with formation of ferrite. And at the temperatures lower than line *SE secondary* cementite emerges from austenite. In the GSP field of the diagram a mixture of ferrite and austenite is present. Below line GP there is only ferrite. Under the further cooling to the temperatures corresponding to line PQ, *tertiary* cementite appears from ferrite.

In the point S at 727 °C all austenite containing 0.8 wt% C breaks up and turns to a mechanical mixture of ferrite and cementite called *pearlite*. In any alloy of the system at the temperatures corresponding to the line *PSK* (the line of eutectoid transformation), the rest austenite transforms into a pearlite.

On the state diagram iron–carbon the alloys which are taking place more to the left of the point E (carbon content less than 2.14 %), are called *steels*. The alloys which are taking place more to the right of the point E (carbon content from 2.14 wt% to 6.67 wt%), are called *cast irons*.

The optical microstructural analysis

The metallographic analysis of metals and alloys is the research of structure of materials at the large magnification with the help of a microscope, and the structure observed is called *a microstructure*. Investigation of metal structure using microscope is possible only if reflection of light beams from a researched surface is intensive enough. Therefore the surface of a sample should be specially prepared. For sample preparation it is necessary to cut out a sample from the researched metal, to polish its surface, and then to etch it. The method of selective dissolution of phases is the most widely spread. The method is based on the distinction of physical and chemical properties of separate phases and grain boundaries. As a result of various intensity of the dissolution the relief of a sample surface is created. This relief disseminates or reflects a light beam in a microscope, making the structure visible. Samples of carbon steels and cast irons are usually etched with 3–4 % HNO₃ solution in spirit.

Microstructure of carbon steels

Alloys based on iron with carbon content up to 2 wt%, and also metallurgical impurities (Si, Mn, S, P) are called *plain carbon steels*.

Alloys containing up to 0.02 wt% C are classified as *a commercially pure iron*. Steel with carbon content 0.8 wt% C is called *an eutectoid steel*. Steels containing from 0.02 up to 0.8 wt% C are *hypoeutectoid steels*, and ones containing more than 0.8 wt% C are *hypereutectoid*.

Depending on a class it is possible to distinct four types of structures in steels.

The first type of structure is *ferrite and tertiary cementite*. It is observed in technical iron. Under microscope ferrite is observed as light grains of unequal brightness (fig. 2a). Tertiary cementite is situated inside the grains of ferrite as the smallest particles.

The second type of structure is a *pearlite*. It is observed in eutectoid steel.

Pearlite in most cases has a lamellar structure. Dark stripes, which are seen in pearlite, represent shadows on ferrite surface, produced by projecting after etching cementite areas (fig. 2b). Alongside with a lamellar structure, pearlite may have a granular one (fig. 2c).



Fig. 2 Microstructures in steels: commercially pure iron (*a*), lamellar pearlite (*b*) and granular pearlite (*c*) (?400)

The third type of structure is *a ferrite and pearlite*. It is observed in hypoeutectoid steels. Under cooling of a hypoeutectoid steel ferrite is the first structure appeared after diffusion decomposition of austenite. Light grains of ferrite are formed on boundaries of austenite grains. The rest austenite transforms to pearlite grains at the eutectoid temperature.

The more the concentration of carbon in hypoeutectoid steel is, the less the amount of ferrite grains is, and the more amount of pearlite areas becomes (fig. 3a, b). In the alloys containing 0.5–0.75 wt% C ferrite is situated on the grain boundaries of pearlite as the separate grains (fig. 3c).



Fig. 3 Microstructures in hypoeutectoid steels with carbon content of: a - 0.1 wt%; b - 0.3 wt%; c - 0.7 wt% (?250)

The fourth type of structure is *a secondary cementite and pearlite*. It is observed in a hypereutectoid steel. Upon cooling of the hypereutectoid steel, cementite is the first structure appeared after diffusion decomposition of austenite. Secondary cementite emerges firstly out of austenite and forms thin envelopes on boundaries of austenite grains. After pearlite transformation each pearlite area appears surrounded with a brittle cementite envelope. In a microscope cementite looks like a light inseparable net dividing lamellar pearlitic areas (fig. 4).

If the excess the light phase (ferrite or cementite) occupies less than 20 % of the etched sample area, a presence of some experience makes it possible to distinguish secondary cementite from ferrite due to their shape and color. The net of excess the

ferrite after annealing is made of separate compact particles while secondary cementite looks like almost continuous light net. Besides, the net of secondary cementite erects above pearlite as a relief. At last, cementite seems under a microscope lighter in comparison with ferrite.



Fig. 4 Microstructure of hypereutectoid steel with carbon content of 1.2 wt% (?300)

Microstructure of cast irons

Alloys based on iron with the carbon content from 2 up to 6 wt% and as with more, than in steel, amount of metallurgical impurity (Si, Mn, S, P) are called *cast irons*.

According to the structure they distinguish cast irons in which carbon forms a chemical compound with iron (cementite Fe_3C), and cast irons in which carbon, basically, is in a free condition, as graphite.

The first type is called *white cast iron*. White cast iron can be produced under rapid cooling when casting details and work pieces.

The structure of white cast iron is described by the right part of the iron–carbon diagram (fig. 1). According to the diagram, there exist three types of white cast iron: *hypoeutectic* with 2.14–4.3 wt% C, *eutectic* with 4.3 wt% C, and *hypereutectic* with more than 4.3 wt% C.

Hypoeutectic cast iron has a structure consisting of pearlite, ledeburite and secondary cementite, eutectic cast iron has ledeburitic structure and hypereutectic cast iron consists of ledeburite and primary cementite (fig. 5).



Fig. 5 Microstructure of white cast irons: a – hypoeutectic; b – eutectic; c – hypereutectic (?500)

Cast irons, which contain free carbon (graphite), are classified according to the form of graphite inclusions:

1. Grey cast iron. It contains graphite as lamellar inclusions or flakes (fig. 6*a*). Grey cast iron can be received at slow cooling under casting, and also at the increased contents of silicon and carbon.

2. Malleable cast iron with cluster inclusions of graphite (fig. 6b). Malleable

cast iron may be received from white cast iron by special annealing. It is long thermal processing at which white cast iron is slowly heated up to temperatures 950-1000 °C and after certain exposure is slowly cooled. At such annealing cementite transforms into graphite which forms cluster inclusions.

3. *High-strength (or ductile) cast iron* in which graphite has a spherical form (fig. 6*c*). High-strength cast iron is received by its modifying when melted with small amount of magnesium or cerium (about 0.05 wt%). Modifiers promote formation of spherical inclusions of graphite.

The metal basis of these cast irons can be pearlitic, ferritic or mixed of both (fig. 7).



Fig. 6 Structures of cast irons:

a – grey cast iron with ferritic and pearlitic basis; b – malleable cast iron with ferritic basis; c – high-strength cast iron with ferritic and pearlitic basis (?300)

Structure of	Type of cast iron and graphite shape					
metallic basis (hardness)	Grey with lamellar graphite	Malleable with claster graphite	Ductile (nodular) with spherical graphite			
Pearlite (250 HB)	X					
Pearlite and ferrite (200 HB)	The second					
Ferrite (150 HB)						
δ,%	0.2-0.5	5-10	10-15			

Fig. 7 Schemes of cast irons structures

Graphite inclusions affect mechanical properties of metal negatively, especially

plasticity. The less ramified form they have, the less their negative influence is. The most harmful, from the point of view of mechanical properties, the lamellar shape of graphite is (thus plasticity is the lowest), and the most optimum is the spherical form of inclusions providing the maximal plasticity (see Table 1).

Table	1
-------	---

Grade of cast iron		σ_T , MPa (kg/mm ²)	δ, %	HB
	СЧ10	100 (10)	_	190
~ .	СЧ15	150 (15)	_	210
Grey iron	СЧ20	200 (20)	_	230
	СЧ35	350 (35)	_	275
Malleable iron	КЧ 30-6	300 (30)	6	100-163
	КЧ 35-10	350 (35)	10	100-163
	КЧ 45-7	450 (45)	7	150-207
	КЧ 60-3	600 (60)	3	200-269
	ВЧ 35	350 (35)	22	140-170
Ductile iron	ВЧ 40	400 (40)	15	140-202
	ВЧ 50	500 (50)	7	153-245
	ВЧ 70	700 (70)	2	228-302

Russian grades and mechanical properties of cast irons

The note: For grey cast irons the thickness of a casting wall is 15 mm, for malleable cast irons the size of casting is 16 mm.

Test questions

- 1. What is carbon content in eutectoid steel?
- 2. What crystal lattice has α iron and γ iron?
- 3. Give the definitions to the following structures: austenite, ferrite, pearlite, cementite.
- 4. Specify the structures in each field of a steel part of iron-carbon state diagram.
- 5. What processes proceed in steel upon cooling at the temperature 727 °C?
- 6. What is the maximum carbon content in austenite?
- 7. What is the maximum carbon content in ferrite?
- 8. How does the carbon content influence steel properties?

Problem tasks

1. Choose a material for manufacture of shot for shot blasting machine of parts clearing. The shot upon service should not be deformed and should have high hardness and wear resistance. Describe structure of the chosen material.

2. A machine tool bed is made by casting with the subsequent machining. In the process of work the bed does not test shock loadings. Service conditions are easy enough. Choose a material for its manufacture, and describe the structure.

3. Cases of reducers are made of cast iron by casting with the subsequent machining. The material should posses tensile strength $\sigma_T = 500$ MPa, relative elongation 1.5 % and have hardness *HB*230. Choose and substantiate grade of cast iron, and explain its structure.

4. Why white cast irons are applied restrictedly in mechanical engineering? Give the detailed explanation. What types of white cast irons exist, and what are their structures?

5. There was a breakage of a crankshaft of the diesel engine. After microstructure research the conclusion has been drawn that the structure of the given alloy consists of pearlite grains with inclusions of lamellar graphite. On specifications the given material should possess $\sigma_T \ge 650$ MPa, $\delta \ge 2$ %, $HB \ge 220-300$. What material the crankshaft has been made of? Because of what there was a breakage, and what you recommend for prevention of shaft destruction further?

6. Choose a material for the case of the small electric motor. Working conditions are easy, loadings are small. The case is cast with the subsequent processing by cutting. Explain the structure of cast iron chosen.

7. For mining of gravel from the river suction-tube dredger is used. Turning joints of pipes for gravel transportation are made of cast iron. Service conditions: the big hydroabrasive wear, shock loadings, constant vibration. Choose and prove cast iron grade.

8. Choose a material for manufacture of heating batteries. A way of their manufacture is casting. Explain the structure of chosen alloy.

LABORATORY WORK № 5

THERMAL TREATMENT OF CARBON STEELS

The purpose of the laboratory work

- 1. To study theoretical bases of quenching and tempering of carbon steels.
- 2. To study the influence of the carbon contents on hardness of steel after quenching.
- 3. To study the influence of the cooling medium (cooling rate) on steel hardness after quenching.
- 4. To study the influence of the tempering temperature on steel hardness.

The equipment and materials

- 1 Heating furnaces with automatic devices for temperature regulation.
- 2 Tanks with various cooling liquids (water, oil).
- 3 Rockwell hardness tester with a diamond cone.
- 4 Samples of carbon steels with various contents of carbon.

Basic theoretical principles

Properties of alloy depend on its structure. The basic way, allowing to change structure, and, hence, properties of an alloy, is thermal treatment, or heat treatment. Thermal treatment represents a set of operations of heating, exposure and cooling in order to change an internal structure of an alloy and to receive required properties. The basic kinds of thermal processing are annealing, normalizing, quenching and tempering.

Annealing and normalizing are applied so that to eliminate defects of the previous technological operations (processing by pressure, casting, welding) and to prepare structure and properties of a material for the subsequent technological procedures. They improve ductility of steel and its machinability upon cutting, and produce microstructure favorable for final thermal processing. Heating for *annealing*, depending on purpose, can be lower or higher than critical temperatures (the temperatures corresponding to phase transformations). *The cooling is very slow and performed together with the furnace*. The results of annealing are the following: chemical heterogeneity is completely or partly eliminated, internal stresses decrease, the grain size is fine, plasticity and toughness rise, strength and hardness are reduced, machinability by cutting improves.

Upon *normalizing* steel is heated up to *austenitic* state (30-50?) above the line *GSE* according to the diagram Fe–Fe₃C), left in a furnace for heating through all cross-section of detail and finishing of phase transformations, and then *cooled in air*. As a result the coarse-grained structure becomes fine, eutectoid gets thinner layers and the rough cementite net is eliminated in hypereutectoid steels. It results in improvement of machinability by cutting and ductility of steel, increase of resistance

to brittle fracture. For low-carbon steels normalizing is applied instead of annealing as more economic procedure. Strength and hardness of normalized medium- and high-carbon steels become higher in comparison with annealed one. Normalizing is applied as an intermediate operation improving the structure, but sometimes, if it provides satisfactory mechanical properties, – as the final one. For example, large-sized steel castings are subjected to normalizing in order to improve their properties. High-quality shapes – rails, channels, pipes, etc. – are used after normalizing, too.

Quenching of steel is the thermal procedure consisting of heating up to the temperature above the lines of phase transformations, exposure for finishing the transformation and following cooling fast enough to suppress diffusion processes. Thus supersaturated solid solution of carbon in α -Fe – martensite – is received.

The purpose of quenching of steel is to obtain the maximum strength and hardness. Supersaturation of solid solution causes strong distortions in a crystal lattice, which result in big stresses and occurrence of dislocations. The more the carbon content in the martensite is, the stronger the distortions in a crystal lattice are, and the more density of dislocations is, therefore the strength and the hardness of steel is higher, but plasticity is lower (fig. 1).



Heating temperature for quenching is chosen according to the diagram Fe– Fe₃C (fig. 2). For hypoeutectoid (constructional) steels the heating temperature should be higher than the point Ac_3 (line GS), but this excess should not be large:

 $t = Ac_3 + (30-50)$ %C.

Upon such a heating the transformation of ferrite and pearlite into austenite (i. e. full phase transformation) will occur. The subsequent cooling with the rate exceeding the critical one will allow obtaining homogeneous martensite. Excess of optimum temperature causes

growth of austenite grains. Large crystals of martensite with the increased brittleness will turn out from coarse-grained austenite. This kind of defect is called *overheating*. Overheating can be corrected by repeated heating up to optimum temperature Ac_3 + (30–50) % at which full phase transformation occurs.

Heating temperature for quenching of hypereutectoid (tool) steels is carried out up to temperatures:

$$t = Ac_1 + (30-50)$$
 %C.

After being heated up to these temperatures the structure of steel consists of austenite and cementite (i.e. incomplete phase transformation occurs). After being cooled with the rate exceeding the critical one the structure consists of hard and wearresistant crystals of martensite and crystals of cementite, still having the big hardness and wear resistance. Heating to higher temperatures will not result in increase of hardness, but will sharply increase the size of austenite grains, that will negatively affect mechanical properties. The area of optimum temperatures for heating steels upon quenching is shown in fig. 2 (shaded).



Fig. 2 The state diagram Fe–Fe₃C with a range of heating temperatures for hipoeutectoid and hipereutectoid steels

The minimum speed of cooling at which all austenite might be overcooled up to temperature M_s and turned into martensite, is called a critical cooling rate V_{cr} . It can be determined using the diagram of isothermal transformation of overcooled austenite. These diagrams are designed for all steels. Geometrically the critical cooling rate is a tangent to a curve of the beginning of the diffusive transformation of austenite into a mixture of ferrite and cementite. In fig. 3 the diagram of isothermal transformation (or the S-shaped diagram) for steel containing 0.8 % of carbon is submitted. For carbon steels time before the beginning of the diffusive decomposition of austenite τ_{inc} is extremely short (0.5–1 s). Therefore, critical cooling rate is achieved, only if steel is cooled in water or in water solutions of salts. If cooling rate is more than critical one $(V > V_{cr})$, transformation begins at the temperature M_s (the temperature of martensite transformation beginning, or start) and finishes at the temperature M_f (the temperature of martensite transformation ending, or *finish*). If the cooling rate is less than critical one, austenite turns into a mixture of ferrite and cementite, having lamellar structure, which depending on the distances between lamellae is called pearlite, sorbite or troostite. Upon slow cooling (V_l) austenite will turn into pearlite (a rough mix of crystals of ferrite and cementite). With the increase in cooling rate (V_2 and V_3) the number of the centers of ferrite and cementite nucleation increases, and the sizes of crystals of these phases decrease. More disperse,

i.e. fine-grained structures (sorbite and troostite), have higher hardness, than pearlite has.



Fig. 3 The diagram of isothermal decomposition of overcooled austenite (steel containing 0.8 % C)

Tempering is a heating of quenched steel to temperatures below critical point for getting required service properties.

Supersaturated solid solution of carbon in α -Fe (*martensite*) has the large amount of free energy and consequently is not stable. Hence, in the guenched steel the processes resulting in a more stable state of the system should proceed, i.e. carbon should leave the lattice of martensite. Upon heating the speed of diffusion increases: the higher the temperature, the

more the mobility of carbon atoms is. Thus, there is a decomposition of supersaturated solid solution and it leads to the formation of equilibrium phases: carbide and ferrite.

Three types of tempering may be distinguished:

1) low-temperature tempering – from 160 up to 200 %;

2) medium-temperature tempering – from 350 up to 450 %;

3) high-temperature tempering – from 500 up to 600 %.

The temperature of tempering is chosen, taking into account required service properties of a certain part (fig. 4).

Low-temperature tempering is applied to details, which are required to be rather hard and wear resistant. Low tempering is appointed for increase of toughness and plasticity of steel without appreciable reduction of hardness. At such a tempering the process of carbon diffusion out of martensite is not completed; carbon atoms form very fine particles of carbide Fe_3C (i. e. martensite becomes less strained). This structure refers to *tempered martensite*. Thus a reduction of internal stresses takes place. This type of tempering is applied, basically, for cutting and measuring tools, for different parts after cementation.

Medium-temperature tempering is applied to parts that must have high elastic properties. At temperature of this tempering all excessive carbon leaves α -Fe lattice and forms fine cementite crystals, which have a needle-like shape. It means that martensite decomposes into a mix of cementite and ferrite. This cementite and ferrite mixture in which cementite has a small-needle shape, refers to *tempered troostite*. As

a result, brittleness sharply decreases, tensile strength is reduced, but the yield strength rises dramatically. This type of tempering is applied to springs and other details which work at fluctuating loadings and should restore quickly their shape after deformation. Usually for manufacturing elastic elements steels with the carbon contents from 0.5 up to 0.7 %, both carbon, and alloyed are used. These structural steels are presented in a special group of spring steels.

High-temperature tempering is applied to parts of the machines subjected to complex loading: static, dynamic and fluctuating ones. After high tempering the structure consists of ferrite and rather large cementite crystals having spherical shapes. This microstructure is called *tempered sorbite*. High tempering provides the best combination of strength, plasticity and impact toughness; internal stresses are fully removed.



Fig. 4 Influence of tempering temperature on mechanical properties of the quenched carbon steel

Quenching in a combination with high tempering is called *a heat refining*, or *a toughening*. The special group of constructional steels is exposed to such kind of processing (with the contents of carbon from 0.3 up to 0.5 %), having the name *heat-hardenable steels*. After such a heat treatment steel has higher characteristics of strength (*TS* and σ_y), plasticity (δ and ψ) and toughness (*KCU*) in comparison with steel after annealing. That is why a name "improvement" appeared – mechanical characteristics of steel are improved.

Tasks and methodical instructions

1. Determine the contents of carbon in steels due to designation of grades and choose the optimum temperature of heating for the given steels using the state diagram "iron – iron carbide".

2. Calculate the time of heating of samples in the furnace in dependence of

temperature of heating and the sizes of samples.

3. Determine experimentally the cooling rate providing reception of martensite structure. Cool the samples heated up to temperatures of quenching, in various mediums: in water, in mineral oil and in quiet air. Then measure hardness by the Rockwell method. Put the results of the experiment into the table 1.

					I dole 1
		Quenching	order		
Steel grade	Temperature of heating <i>t</i> , °C	Time of heating τ , min	Cooling medium and <i>V</i> , °C/s	Hardness, <i>HRC</i>	Microstruc- ture
1020			water, 600 °C/s		
1045			water, 600 °C/s		
10120			water, 600 °C/s oil, 150 °C/s air, 30 °C/s		

4. Draw the plots according to the results of the laboratory work:

a) dependence of steel hardness after quenching on the carbon contents;

b) dependence of quenched steel hardness of grade 10120 on the cooling rate.

5. Subject the quenched samples of various carbon steels to tempering at temperatures 200, 400 and 600 $^{\circ}$ C. Accept time of tempering is equal to 0.5 hour. Cool samples after tempering on air.

6. Determine hardness of samples after tempering using the Rockwell tester and put the results into the table 2.

Table 2

Table 1

	Hardness	Tempering	Heating time	Hardness	
Steel	after	temperature	τ , min	after	Microstruc-
grade	quenching,	<i>t</i> , °C		tempering,	ture
	HRC			HRC	

7. Draw the plots of dependence of hardness on temperature of tempering for all steels investigated.

8. Make conclusions.

Test questions

1. For what purpose is steel quenching carried out?

2. In what temperature range sorbite is formed?

3. What results increase in heating temperature of hypoeutectoid steel under quenching from $(Ac_1 + 50^\circ)$ to $(Ac_3 + 50^\circ)$ in?

4. What is the difference among structures: pearlite, sorbite and troostite?

5. What dispersion degree (the grain size) of diffusion transformation products depends on?

6. What is the type of martensite crystal lattice?

7. What is the difference martensite from austenite of which it was formed?

8. What the temperature of steel heating for quenching depends on?

9. What is the difference between the structure of steel 10120 after quenching from the temperature above Ac_1 and from the temperature above Ac_m ?

10. How increase in carbon content influences the temperature of heating for quenching?

11. What hardenability of steel (hardness after quenching) depends on?

12. How properties of the quenched steel change at increase in the carbon content to 0.8 wt%?

13. Give the reason of high hardness and strength of steel after quenching.

14. Explain, why structural steels are not quenched from the temperatures above Ac_1 ?

15. Why it is necessary to cool steel with a speed exceeding the critical one?

16. What means the critical cooling rate?

17. Describe the characteristic features of martensitic and pearlitic transformation.

18. For what purpose is tempering of steel after quenching carried out?

19. For what parts low-temperature tempering is applied?

20. For what parts medium-temperature tempering is applied?

21. For what parts high-temperature tempering is applied?

22. What processes proceed in steel structure upon tempering?

23. What the structure tempered martensite represents?

24. How properties of the tempered steel change with growth of tempering temperature?

25. What is the difference between sorbite and troostite after tempering?

26. What operation is it necessary to carry out, if after tempering hardness *HRC* and strength σ_T are lower, than it is required?

27. What operation is it necessary to carry out, if after tempering hardness *HRC* and strength σ_T are higher, than it is required?

28. What temperature of tempering should be chosen for parts which must have high elastic properties?

29. What temperature of tempering should be chosen for parts which must have high hardness and wear resistance?

30. What will occur in steel structure if after tempering at 600 °C make additional tempering at 200 °C?

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