FSBEO HE
"UFA STATE AVIATION TECHNICAL UNIVERSITY"

Department of Material Science and Physics of Metals

LABORATORY WORKSHOP

"MATERIAL SCIENCE"

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Content

THE STUDY OF THE CRYSTALLIZATION PROCESS AND STRUCTURES OF CAST METAL

1. PURPOSE AND TASK OF LABORATORY WORKSHOP

1.1. To study the process of crystallization of salt solutions: describe the sequence of crystallization on the example of a solidifying drops of salt solution.

1.2. To study the influence of external factors on the process of structure formation during crystallization.

1.3. Get acquainted with the structure of the cast metal and identify differences in its structure depending on the conditions of crystallization.

2. THE BRIEF OF THEORETICAL PART
2.1. The process of the crystallization

The crystallization is the process of solid crystals forming from a liquid or gas. The transition of a substance from one state of aggregation to another is a phase transformation. *The phase* is chemically and physically homogeneous part of a substance (or system), separated from its other parts by the interface, when passing through which the composition, structure or properties change explosively.

The crystallization process, like any phase transformation, occurs spontaneously. *The driving force* of crystallization is a decrease in the free energy of the system. If the system can exist in the liquid and crystalline states, the relationship of free energy to temperature can be represented graphically (Figure 1). Curve 1 describes the dependence of the free energy on temperature for the liquid phase, and curve 2 – for the crystalline phase. The temperature T_o is one at which the free energies of liquid and solid states are equal is *the equilibrium temperature*. At this temperature, both phases are stable; therefore, crystallization cannot begin, as there is *no driving force*. Crystallization will begin when the free energy of the crystalline phase is less than the free energy of the liquid phase. In this case, a spontaneous decrease in energy by the value of $\Delta F = F_{liq} - F_{crys}$ per unit volume of the substance will occur. This gain of the free energy ΔF is *the driving force* of the crystallization process. The difference between the equilibrium temperature T_o and the
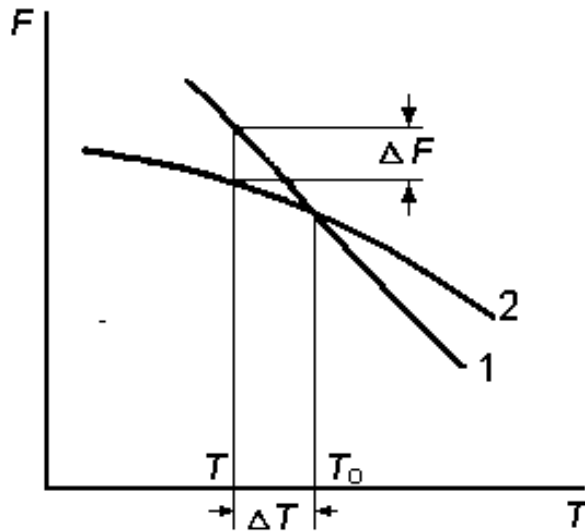
Figure 1. The dependence of the free energy $F = U - TS$ on temperature
for the liquid and crystalline phase of matter

actual temperature $\Delta T = T_0 - T$ is called *the degree of supercooling*. In the presence of supercooling in the substance, the nucleation of crystallization centers can begin.

The crystallization process consists in the formation (*nucleation*) of crystallization centers and the growth of crystals from the formed centers. The process kinetics is quantitatively described by *the rate of nucleation of crystallization centers* (RNC) – the number of crystallization centers occurring in a unit volume per unit of time, and *the linear growth rate of crystals* (LGR) - the rate of movement of some the plane of crystal is parallel to oneself. Let us consider these two processes in more detail.

2.1.1. The nucleation of crystallization centers

At any temperature, groups of atoms with an ordered structure are formed in the liquid. These groups are unstable; they break up and re-formed in different parts of the liquid phase. When cooling the liquid phase below T_0 temperature, then some of the largest of them can become stable. They are spontaneously *nucleation center* or *crystallization centers*. However, not all nucleus can grow. Only crystals of sufficiently large size – *a – critical size* – become capable of growth.

Let one crystallization center be born in the system in the form of a cube with an edge *a*, this will lead to a decrease in the volume free

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energy

$$\Delta F_{vol} = -V\Delta F, \tag{1}$$

where V - the volume of the crystal is equal to a^3. The crystal formation is accompanied by the occurrence of an interface between the liquid phase and the crystal, and the work expended on its creation is equal to

$$\Delta F_{surface} = S\sigma, \tag{2}$$

where S – the surface area of the crystal ($6a^2$), σ–specific surface energy at the boundary of the liquid and crystalline phases (***surface/boundary tension***).

The total change in the free energy of the system will be

$$\Delta F` = -\Delta F_{volume} + \Delta F_{surface}, \tag{3}$$

or

$$\Delta F` = -a^3\Delta F + 6\,a^2\sigma. \tag{4}$$

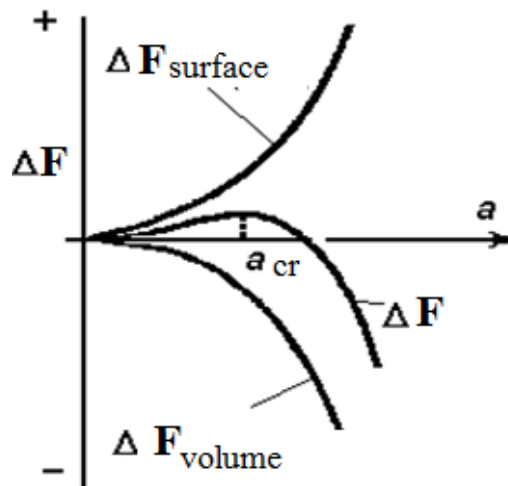In Figure 2 graphic representation of equation (3) is presented: curves of changes in bulk and surface free energy.

Figure 2. Change in free energy upon formation of the nucleus

For a given temperature T the values of ΔF and σ are constant, and $\Delta F`$ depends on only the size of the crystal. Differentiating equation (4) get:

$$\frac{d\Delta F'}{da} = -3a^2 \cdot \Delta F + 12a\sigma \tag{5}$$

It follows that $\Delta F`$ has a maximum at

$$a_{cr} = \frac{4\sigma}{\Delta F'} \tag{6}$$

Crystal of size a_{cr} called a *critical nucleus size.* It is in unstable equilibrium with the initial phase**:** its growth and decay both cause a decrease in the free energy of the system. Crystals larger than a_{cr} capable of growth, while the free energy of the system will decrease.

One can see from (6), that a_{cr} magnitude depends on ΔF, while ΔF depends on the *degree of supercooling* (Figure 1). Therefore, the greater the *degree of supercooling*, the smaller a_{cr} and, accordingly, the smaller the critical value of the free energy (Figure 3) and the smaller the size of the critical nucleus capable of growing.

The work of critical size nucleus formation is ensured by the energy fluctuations, and this process is called *homogenous nucleation.* The greater the degree of supercooling, the smaller is nucleus critical size and smaller its formation work. Therefore, with an increase in the degree of supercooling, the number of nucleus of a critical size arising in a unit volume per unit of time increases. Their number is proportional to *exp(–$\Delta F_{cr}/kT$).*
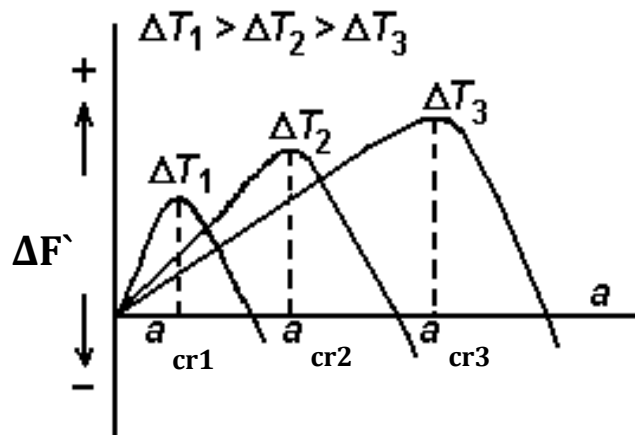

Figure 3. Changes in the free energy of the system depending on the size of the formed crystal

The critical nucleus size becomes the center of crystallization when atoms from the liquid phase begin to attach to it by diffusion. With a sharp decrease in temperature, i.e. with significant degrees of supercooling, this process slows down. At temperatures when diffusion processes are suppressed, crystallization centers are not formed, and the substance passes into an amorphous state. Thus, as supercooling degree increase, R.N.C. first increases, passes through a maximum and decreases. The degree of supercooling depends on the nature of the metal, its purity and the cooling rate. Theoretical calculations show that the maximum values of R.N.C. are achieved at ~ 0,2 T_o. Below are the

ΔT values of some metals with homogeneous nucleation:

Metal	Pb	Sn	Sb	Ag	Cu	Fe	Ni	Co	Pt
$T_{melt,}$ °C	327	232	630	962	1083	1539	1452	1494	1773
ΔT	80	118	135	230	236	295	310	330	370

As can be seen, the values of **ΔT** are rather large, which makes homogeneous nucleation unlikely.

2.1.2. Crystal growth

Crystals can grow by simultaneously joining a certain group of atoms to any plane. Such a group of atoms is called a *two-dimensional nucleus*. Consequently, crystals growth rate is determined by the probability of the nucleation of a two-dimensional nucleus and the probability of its attachment to the crystal due to diffusion. From this it follows that the degree of supercooling affects the linear growth rate (L.G.R.) in the same way as it affects the rate of nucleation of the centers (R.N.C.). It was established experimentally that the maximum L.G.R. most often corresponds to lower degrees of supercooling than the maximum of the R.N.C. (Figure 4).
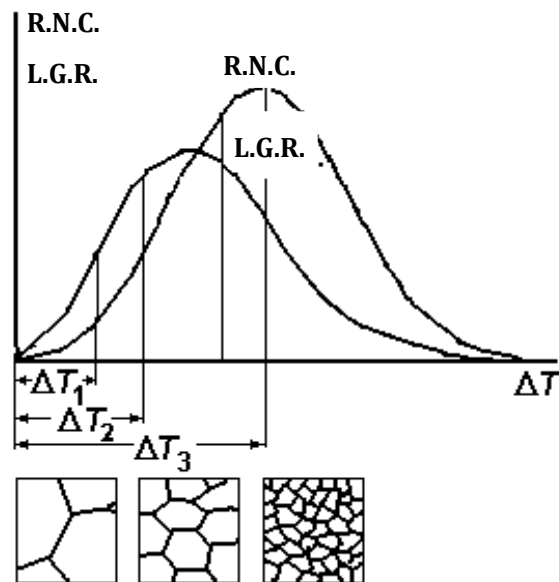

Figure 4. Dependence of the parameters of crystallization and the final microstructure on the degree of supercooling

The curves in figure 4 are called ***Tamman curves***, who obtained them experimentally in 1935 for the first time. Based on their analysis,

one can conclude that L.G.R. and R.N.C. are a function of the supercooling degree, which in turn depends on the cooling rate – the greater the cooling rate, the greater the degree of supercooling that can be achieved. Therefore, by changing the cooling conditions, it is possible to control the crystallization process. The final grain size is determined by the number of centers and the crystal growth rate.

2.2. Crystallization process influencing factors

As was shown, the work of formation of a critical nucleus size is determined by the magnitude of the surface energy at the boundary between the liquid and crystalline phases. So, all factors affecting the surface energy affect the crystallization kinetics as well. The presence of ready-made interface surfaces facilitates the process of nucleation of crystals. Real metals always contain a large number of impurities, the smallest particles of insoluble phases, oxides, which serve as ready interfaces. This leads to a strong acceleration of nucleation and crystallization as a whole. The crystallization in the presence of ready interfaces is called *heterogeneous nucleation*. The walls of the crystallizer into which the liquid metal is cast are also used as the ready interface. The presence of heat removal through the walls of the crystallizer contributes to an increase in the degree of supercooling.

A number of mechanical properties of metals: strength, ductility, endurance limit stress, impact toughness are higher at small grain size. Therefore, in the practice of manufacturing castings, especially if they are not subjected to further deformation treatment, they use *modification* – introducing special additives into the melt, which without sufficient chemical composition changing create additional crystallization centers, contributing to the refinement structure.

The magnitude of the degree of actually supercooling or overheating that is achieved depends on the nature of the metal. It increases with an increase in its purity and cooling rate. Under normal production conditions, the degree of supercooling of metals during crystallization ranges from 10 to 30 °C; at high cooling rates, it can reach hundreds of degrees. Raise of the cooling rate, lowers the temperature to which the liquid metal can be supercooled until the end of crystallization. This position has acquired in recent years not only theoretical, but also of great practical importance. It turned out that with the use of special equipment it is possible to achieve very high cooling

rates (10^7–10^9°C/c), at which the liquid metal can be supercooled to a solid state practically without its crystallization. As a result, the metal has either a *nanocrystalline* structure, or even turns out to be in an *amorphous* or *glassy* state with unusual mechanical and physical properties.

Conversely, if you have only one crystallization center and create crystallization conditions such that supercooling (including concentration cooling) is not created in front of the crystallization front, which could lead to the formation of nuclei of new crystals, a *monocrystal* ingot can be obtained. In some cases, it is even possible to produce crystals with a controlled number of defects.

2.3. Crystals form and size

In real metals and alloys, crystals shape and size depend on a number of factors: the crystal structure, the growth rate, the direction of the heat sink. Usually, crystals form the right geometric shape, corresponding to their crystalline structure. If the growth rate of a crystal is the same in all directions, the crystals grow, maintaining the correct shape, until it is surrounded by a liquid phase on all sides. Under conditions of significant overcooling, the elements of the faceting of crystal will be in a non-identical position with respect to the supply from the liquid phase. The feeding is best fed to the tips, then to the crystal edges, and, finally, to the faces of the polyhedron, which causes uneven crystal growth, leading to the formation of skeletal, needle, spiral, and other forms.

If the growth rate of crystals in one direction is larger, tree-shaped crystals or *dendrites* are formed (Figure 5, *a*). At first, the crystal acquires an elongated shape in the direction of higher growth rate, forming an axis of I order, then begins to grow in lateral directions, and not only along the entire surface, but in separate areas. As a result, axes of II order etc., branch off from the main axis of the first order corresponding to the direction of the highest growth rate. Dendrites grow until they come into contact with each other. After that, the interdendritic spaces crystallize, and the dendrites turn into crystals. Crystals or grains may consist of several equally oriented dendrites. The metal ingots produced under industrial conditions usually consist of three zones (Figure 5, *b*), which is consistent with the dependence shown in Figure 4.
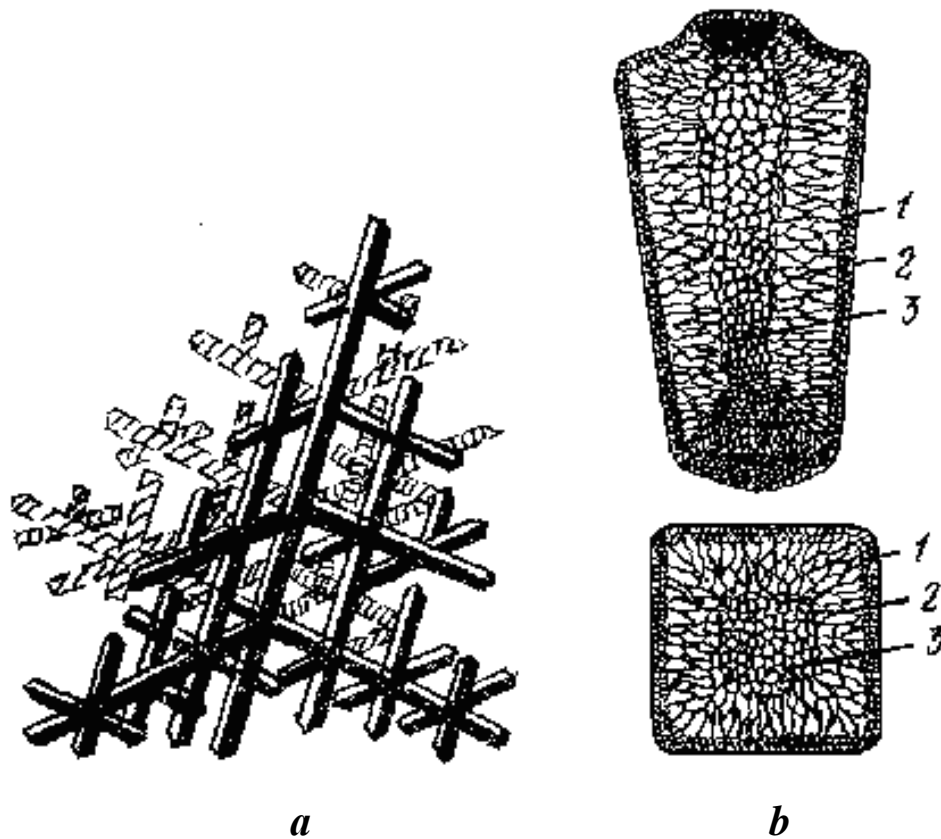
Figure 5. Dendrite growth pattern (a) and metal ingot structure (b):
1– a zone of small crystals, 2 – a zone of column (fringe) crystals,
3 – zone of equiaxed crystals

A high degree of supercooling occurs in the outer layer upon contact of the liquid metal with the walls of the mould, leading to the formation of a large number of crystallization centers, therefore the outer layer has a fine-grained structure. After the formation of the outer layer, the cooling rate, respectively, and ΔT, decreases, the number of centers and the growth rate decrease.

However, an L.G.R. decrease to a lesser extent and in the conditions of a directional heat sink, a second zone is formed – columnar crystals having a dendritic structure. In the center, a ΔT ingot is usually small, there is no directional heat sink, and a zone of large equiaxed grains is formed, which also have a dendritic structure.

3. THE WORK ORDER

To study the crystallization process of supersaturated solutions used salts: sodium chloride $NaCl$, ammonium chloride NH_4Cl, and

ammonium chloride NH_4Cl, modified with $FeCl_3$. The construction of a metal ingot is studied on aluminum alloy ingots obtained by crystallization under normal conditions when the melt is cooled in a water-cooled container, using modification and exposure to ultrasonic vibrations during crystallization.

The order of the work:

a) Drop solutions of sodium chloride salts $NaCl$, ammonium chloride NH_4Cl and ammonium chloride NH_4Cl, modified with $FeCl_3$ on a glass and study the process of nucleation and growth of crystals; sketch the stages of the crystallization process, paying attention to the following points:

- Where does the crystallization process begin?

- What is the shape of the first crystals?

- In which direction the crystallization process continues?

- What is the shape of crystals formed in the second order?

- Where does crystallization end?

- What crystallization zones can be distinguished?

- What are the distinctive features of the structure of solutions 1, 2, 3?

b) Drop of NH_4Cl chloride solution on a glass and in the process of cooling blow on a drop or shake the glass slide slightly. Pay attention to the drop structure changes. Explain your observation.

c) Study and sketch the macrostructure of aluminum alloy ingots, explain the influence of crystallization conditions on the formation of the structure.

4. REPORT REQUIREMENTS

According to the results of the work to make a report, which should include:
- the name and purpose of the work;
- materials of work performance;
- description of salts crystallization sequence;
- a sketch of the crystallized droplets of $NaCl$, NH_4Cl, and NH_4Cl,

modified $FeCl_3$;

• explanation of the sequence of crystallization of the commonalities and differences of the structure drops salts;

• scheme and explanation of the structure of metal ingots;

• the conclusions of the work.

5. TEST QUESTIONS

1) During overcooling in the melt nucleus appeared having a critical nucleus size. What factors can lead to the growth of this nucleus?

2) What is the physical essence of modification? Due to some factors, you can reduce the amount ΔF in equation (3) with modifying?

3) Can the ice begin to melt at 0 $^{\circ}$C. Why?

4) Under what conditions of the cooling you can get nanocrystalline or amorphous ingot?

5) Under what conditions of the cooling you can get monocrystal ingot?

6) How many phases does the water ice system consist of when, along with water, there are five pieces of ice of different shapes and sizes?

References

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DEFINITION OF MECHANICAL PROPERTIES OF STRUCTURAL MATERIALS

Introduction

Ability of details of machines and various constructions to preserve working capacity and non-failure operation depends on properties of materials of which they are fabricated. The knowledge of properties of materials taking into account conditions of a stressing and maintenance of details allows designers to select a material correctly, for the processmen knowing mechanical and technological properties of materials - to assign an optimum condition of their processing, and for the economists having of sufficient knowledge in the field of technique and costs of materials - to calculate economic efficiency of manufacturing techniques of details of machines correctly.

For definition of mechanical properties of materials there are carried out special tests. In this paper the methods of metals tests on a tension, hardness and bending are considered.

Before execution of operation students should independently examine this reference, prepare answers for tests and control questions for self-examination.

At execution of laboratory work students are formed such professional competence as ability to use methods of standard tests for definition of mechanical properties and technological indexes of constructional materials.

Having performed laboratory work, the student should:
• **know:**
- Methods of standard tests of materials for definition of mechanical properties;
- The equipment for standard tests;
• **be able** to apply methods and the equipment of standard tests of materials for definition of mechanical properties;
• **have skills:**
- Operations on test equipment for standard tests;
- Analysis of the received results according to technical requirements.

Before each lesson the student should be prepared for forthcoming operations and should made the preliminary record according to requirements. Records are made on each laboratory work accurately and

competently, in the text it is necessary not to suppose abbreviations of records. Sketches and graphs must be followed with necessary explains. Applied terms and designations should correspond to standard requirements. Before execution of laboratory work the preliminary individual record on operation, and also theoretical readiness of students is checked. The students who do not made the individual record, and not ready to the answer the control questions are not supposed to execution of operations. Upon finishing of laboratory woks students are obliged to return the record for checking and to speak in support of paper, answering questions of the teacher about the contents of laboratory work and results of its practical part.

LABORATORY WORK №2
DEFINITION OF MECHANICAL PROPERTIES OF STRUCTURAL MATERIALS

1. The operation purpose – acquisition by students of skills and the abilities necessary for definition of the basic mechanical properties of metal materials.

2. Operation problems:

1) to study methods of definition of mechanical properties of materials from test on a tension;

2) to master techniques of mechanical properties control via materials hardness gauging;

3) to learn impact strength definition.

3. **A theoretical part**

Strength, elasticity, plasticity, hardness, viscosity are treated to mechanical properties of metals and alloys. These properties are the basic indexes on which suitability of metal to various working conditions is judged. First three properties are defined by tests of materials on a tension.

3.1. Tensile test [GOST 1497 - 84]

For tensile test use special specimens (fig. 1.1) with length of a working part l in the form of the barrel or with a rectangular cross section (flat samples).
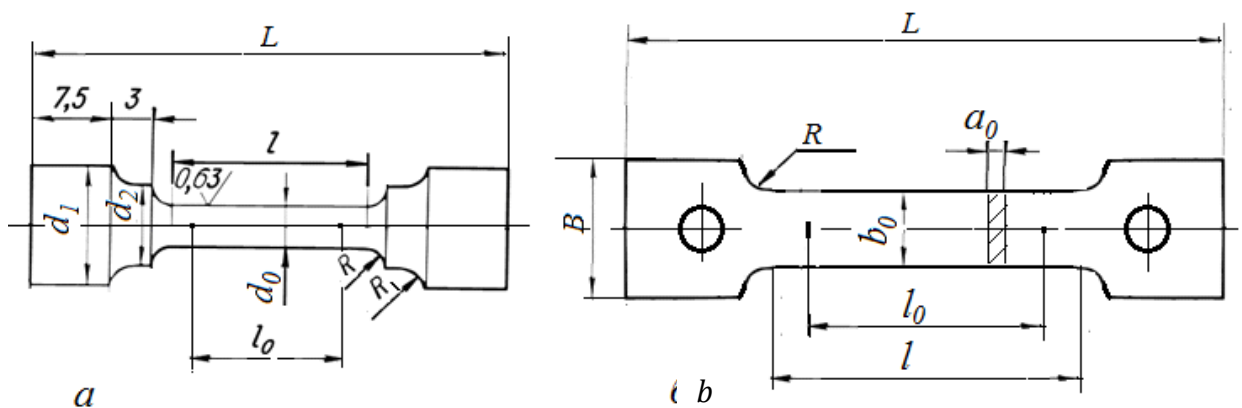

Fig. 1.1. Standard specimens for tensile test: a - cylindrical; b – flat

On a working part of the sample the initial settlement length of the

sample l_0 (mm) is selected by matchmarks. Elongation is defined due to this length. Samples of a circular cross-section with a diameter $d_0 = 5$ mm and $l_0 = 25$ mm are applied most often (fig. 1.1). Flat samples apply at tests of sheet materials. They have following sizes: initial depth a_0 and width b_0, settlement length of a working part l_0 (fig. 1.1). Tests are carried out on universal tensile test machines (look fig. below).

Universal tensile test machine INSTRON

During test tensometric device of the tensile machine records in the form of the chart dependence between axial load P and an increment of length of the sample Δl. On fig. 1.2 typical tension charts recorded by tensile machine during testing of metal materials are presented. The chart with gradual transition from elastic in a plastic deforming region (fig. 1.2.a) is peculiar to the majority of metals in a plastic condition (steel alloys, colored alloys). For low-carbon annealed steels and some bronses after their annealing the chart with a plasticity stage (fig. 1.2.b) is characteristic. Fragile metals (tempered and not drawed steels, cast iron, etc.) fail at small plastic deformations (fig. 1.2.c).
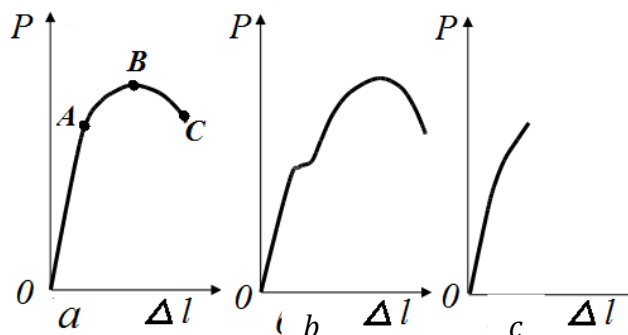

Fig. 1.2. Chart of a tension of various metals: *OA* - area of an elastic strain; *AB* - a site of viscoelastic strains; *BC* - a collapse *stage*

For practical evaluations of engineering stresses there are used the charts of a tension with characteristic points (fig. 1.3). The tension chart $P - \Delta l$ characterizes behavior of the sample made of tested material with certain geometrical parameters. To eliminate influence of the sizes, the chart $P - \Delta l$ is re-counted in the diagram $\sigma - \varepsilon$, where σ - stresses, ε - a relative strain which is defined as: $\varepsilon = (l - l_0)/l_0$, where l - current length of the sample during test. The chart $\sigma - \varepsilon$ - the same diagram $P - \Delta l$, drawn on the scale of P/F_0 on a axis of ordinates and in scale l/l_0 - on a axis of abscissas. At a tension the sample is lengthened, and its cross-section is continuously diminished. But as the area of cross section of the sample into each moment of a strain is difficult to define so strength properties (limit of plasticity and tensile ultimate strength) use conditional stresses. They are evaluated by division of a corresponding load onto an initial cross-section of sample F_0.
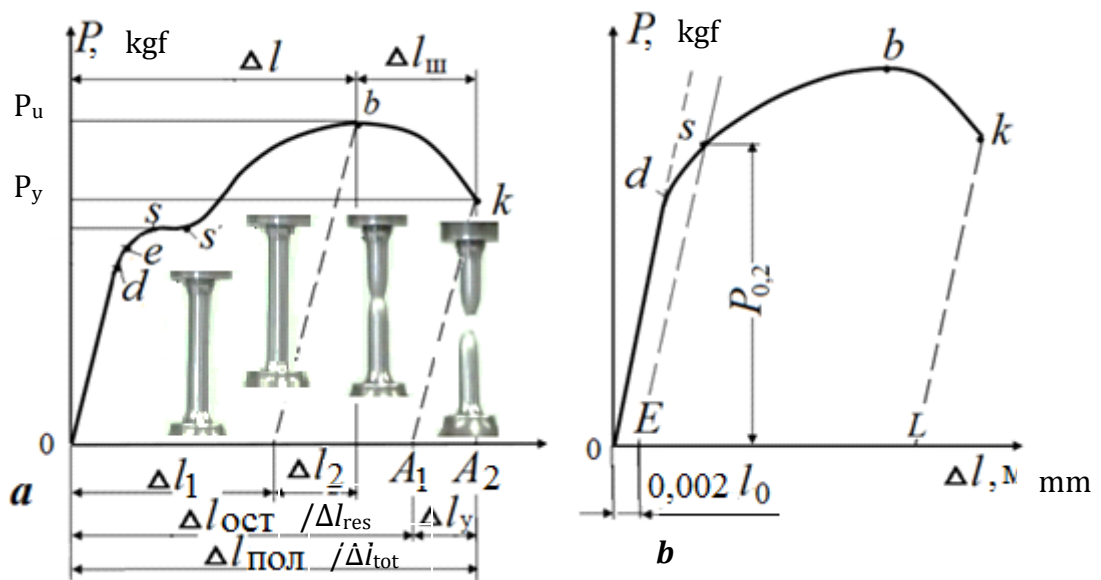

Fig. 1.3. Characteristic sites and points of the chart of a tension:
a - the chart of a tension of a low-carbon steel with a plasticity stage; *b* - the circuit of definition of a conventional yield point $\sigma_{0,2}$ (location of point *S*)

At small loads on a chart site *0d* the residual strain does not arise also the sample after load intakes initial length - its behavior is elastic. This site of an elastic strain has a rectilinear appearance and characterizes rigidity of a material. The smaller elastic strain displays a material under the influence of a load, the higher its rigidity which is evaluated via coefficient of elasticity (Young modulus): $E = \sigma / \delta$, МПа.

At the further increase in a load the rectilinear dependence is

disturbed. Behind a point *«e»* there is an appreciable residual deformation and in a point *«s»* (fig. 1. 3,*)* the plasticity stage **s-s'** is formed. The least stress at which the sample is deformed without appreciable increase in a load ("flows"), is called *as real yield point*. It is defined by the formula

$$\sigma_y = \frac{P_y}{F_0}, \text{MPa} \tag{1.1}$$

For the materials which do not have on the tension chart a horizontal stage (fig. 1.2,*a* or 1.3,*b)*, load $P_{0,2}$ which calls a residual strain equal of 0,2 % from settlement length of the sample is defined. At big enough scale of the primary chart of a tension a value of load $P_{0,2}$ define graphically directly on this chart. For this purpose there postpone a cut equal of 0,2 % (0,002 · l_0, mm) settlement length of the sample and from this point spend a line parallel to an elastic site of the chart. The cross-point of this line with the chart corresponds to magnitude of load $P_{0,2}$ (fig. 1.3,*b)*. When value $P_{0,2}$ is fixed then conventional yield point is evaluated by the formula

$$\sigma_{0,2} = \frac{P_{0,2}}{F_0}, \text{MPa} \tag{1.2}$$

The yield point characterizes stress at which there is a transition from an elastic strain to the plastic.

The stress induced by the greatest load $P_в$ (a point *«b»*) is called *as ultimate resistance* or *tensile strength*:

$$\sigma_u = \frac{P_u}{F_0}, \text{MPa} \tag{1.3}$$

During testing before a point *«b»* the sample has the cylindrical shape. After a point *«b»* there is an appreciable local contraction of the sample with necking where the further strain of the sample concentrates. Therefore on a site ***b-k*** the section of the sample (fig. 1.3,a*)* is quickly diminished, owing to what the stretching load is diminished too. In some point *«k»* the sample is broken off on the least section of neck F_k. In a fragile material necking is not expressed almost, and when tensile

strength reaching σ_B at once there is a collapse (fig. 1.2,c). After test fragments of the sample combine and measure its final settlement length after a break l_K and final diameter d_K (fig. 1.4). Due to it there are defined indexes of plasticity of a material.

Fig. 1.4. The sample before test (a) and after the test, combined for gauging (b)

Two parameters of plasticity are distinguished: an elongation δ and a contraction ratio ψ.

They are defined via formulas

$$\delta = \frac{l_\text{K} - l_0}{l_0} 100\% = \frac{\Delta l_\text{res}}{l_0} 100\%, \tag{1.4}$$

Where l_κ - length of the sample after a break; l_0 - initial settlement length of the sample.

The contraction ratio is evaluated under via formula

$$\psi = \frac{F_0 - F_K}{F_0} 100\% = \frac{\Delta F_K}{F_0} 100\%, \tag{1.5}$$

Where F_κ - the area of sample section after a break;
F_0 - initial section of the sample.

It is conditionally considered that metal is reliable if $\delta \geq 15\%$ and $\psi \geq 45\%$.

Tensile test treats to destroying methods of definition of materials mechanical properties. For current quality inspection of the thermotreated metals usually use not destroying methods the main one from which is hardness gauging.

3.2. Test of metals on hardness

The most simple mode of definition of mechanical properties is hardness gauging. *Hardness* is an ability material to resist to introduction in its surface of solid body – the indenter. As a indenter

there is use the tempered steel ball or a diamond tip in the form of a cone or a pyramid. Methods of impression hardness gauging of Brinnel and Rockwell are most widely used.

3.2.1. Hardness gauging via a method of Brinell. For hardness gauging via a method of Brinell the hardness meter type ТШ is applied. Under load P (from 62,5 to 3000 kg) the steel tempered ball (fig. 1. 5,a*)* with diameter D (1,0; 2,0; 2,5; 5,0 and 10 mm) presses into a surface of a tested material. After load removal on a surface of the sample there is a print (fig. 1.5,c*)*, which diameter d is measured by means of a microscope (magnifier) of Brinell МПБ-2 with accuracy ±0,05 mm (fig. 1.5,*)*. Hardness number define as a ratio of load P to a spherical surface of indentation print F (a spherical segment) - diameter d, left due to the spherical indenter with diameter D:

$$HB = \frac{P}{F} = \frac{2P}{\pi D(D - \sqrt{D^2 - d^2})}. \tag{1.6}$$

In practice hardness do not evaluate via the formula (1.6), and define due to the table of GOST 9012-59, based on the obtained value of print diameter d, and also on known ball diameter D and a value of attached load P.
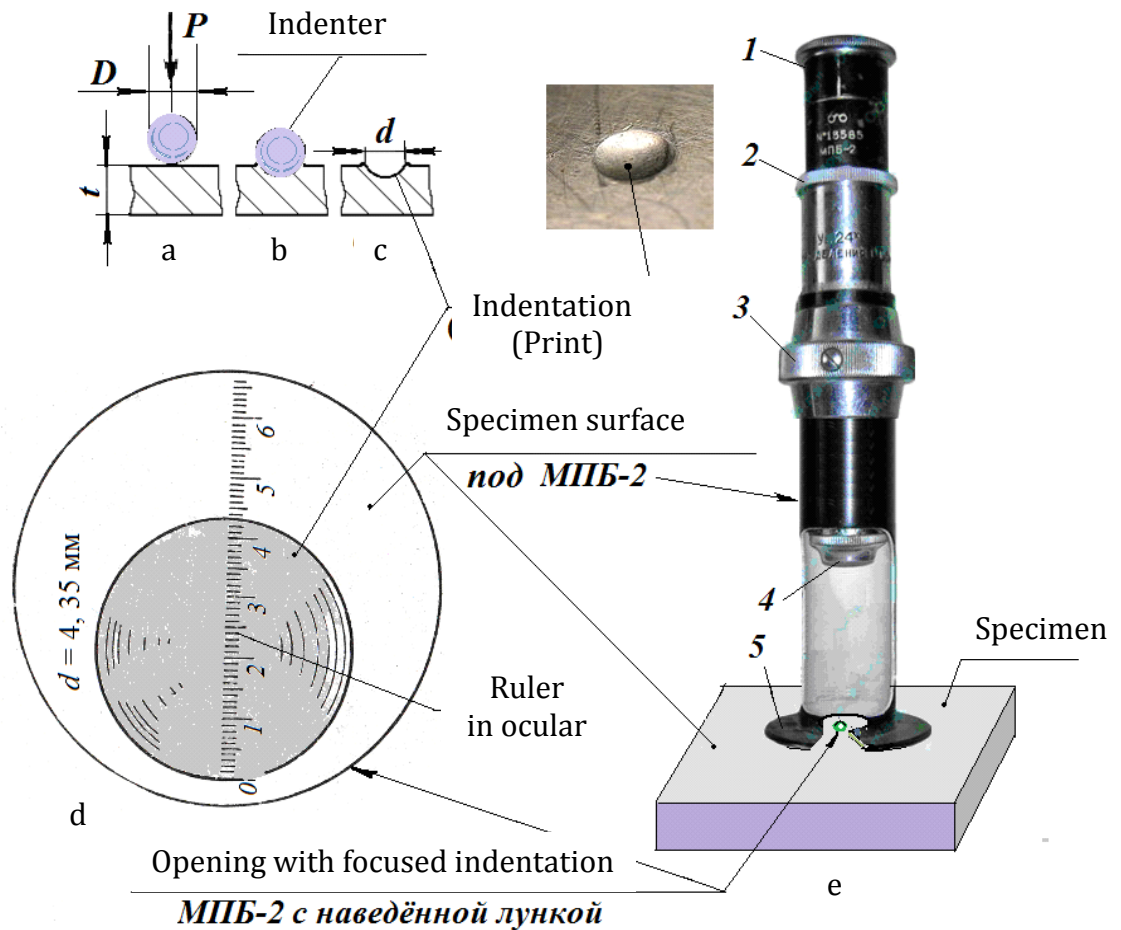
Fig. 1.5. Brinell hardness gauging:
a – beginning of loading; b - end of loading; *c* - the received print (indentation) with diameter *d*; *d* - print gauging; *e* - the magnifier of Brinell adjusted for gauging of diameter of a print

Dimension of hardness via Brinell is kg/mm^2 (10^{-1} MPa). The method is applied to gauging of hardness concerning relatively soft materials (iron, an annealed iron, graphitized cast iron, nonferrous metals and alloys).

Between Brinell hardness number and tensile strength Metals $_{σВ}$, МПа, there are empirical dependences:

• for a steel with hardness   90 … 175 *HB* $σ_u = 3{,}4$ *HB;*
                             175 … 450 *HB* $σ_u = 3{,}6$ *HB*;

• for an annealed copper, yellow metal, bronze $σ_u = 4{,}8$ … 5,5 *HB*;

• for cold-rolled copper $σ_u = 3{,}5$ *HB*;
• for the hammered copper, brass, bronze $σ_u = 4{,}0$ *HB*;
• for tempered duralumin $σ_u = 3{,}6$ *HB*;
• for duralumin after quenching and aging $σ_u = 3{,}7$ *HB*.

Conditions of gauging of hardness (diameter of a ball, a load, stressing time) are selected depending on width of the sample and hardness of the examined material. Thus it is necessary to consider that for deriving of identical hardness numbers at test of the same material with balls of different diameters it is necessary to observe the similarity law between obtained diameters of prints. For sufficing of such law hardness gauging spend at a fixed ratio between magnitude of load P and quadrate of diameter of ball D^2: $K=P/D^2$. This ratio is different for materials with various hardness and it is reached by selection of diameter of ball D, effort P so that diameter of a print was within 0,24 D $\leq d \leq 0,6\ D$.

Brinell hardness is designated by symbol HB (*Hardness Brinell*). At gauging of hardness with use of the tempered steel ball (in order to avoid its collapse) hardness of being gauging material should not exceed 450 units. Then at hardness gauging in the considered conditions ($D=10$ mm; $P=3000$ kg; $\tau = 10$ s) hardness is designated, for example, 300 HB. At use of a ball from a firm alloy it is possible to measure the firmer materials (> 450 HB). In this case hardness is designated, for example, 500 HW. If tests were carried out in the conditions different from considered the record will look like the following: 250 HB 5/750/20 or 500 HB 5/750/20. In these designations of hardness the first digit displays a hardness value (250 and 500); then digits: 5 - diameter of a ball, mm; 750 - loading on indenter, kgf; 20 - time under a load, sec. The method is unsuitable for gauging of hardness of thin sheet materials, details with a firm surface.

3.2.2. A method of hardness gauging via Rockwell. At hardness gauging via a method of Rockwell as a indenter in a device **ТК-2** use a steel ball in diameter of 1,59 mm or a diamond cone with an apex angle 120º. In accordance with GOST **9013-59** depending on a combination «indentor/load» there is distinguished 11 scales of hardness in a method of Rockwell (*A, B, C, D, E, F, G, H, K, N, T*). But most often use scales A, C (with a diamond cone) and B, F (with a steel ball). Rockwell hardness is meant by digits and letters HR with the indicating of a scale A, B, C or F (for example, 60 HRC, 80 HRA and etc.).

At hardness gauging the indenter is pressed into metal by two consecutive stressings (fig. 1.6). In the beginning the indenter is preloaded with $P_0=98$ N (10 kgf) which apply to an exception of influence on gauging result of an elastic strain, a surface finish and local surface damages. The indenter under load P_0 intrudes on distance h_0

from a material surface. On such depth the initial measuring surface is. Then is affixed the basic load P_1 so that the total load makes $P=P_0+P_1$. Under the influence of a total load the indenter will take root into a material on depth h_1 from an initial measuring surface of the sample. Then load P_1 after some soaking is took off and thus indenter **because of** elasticity of the examined material will be displaced up and will stop on distance «*e*» from an initial measuring surface that corresponds to residual depth of indenter permeation.
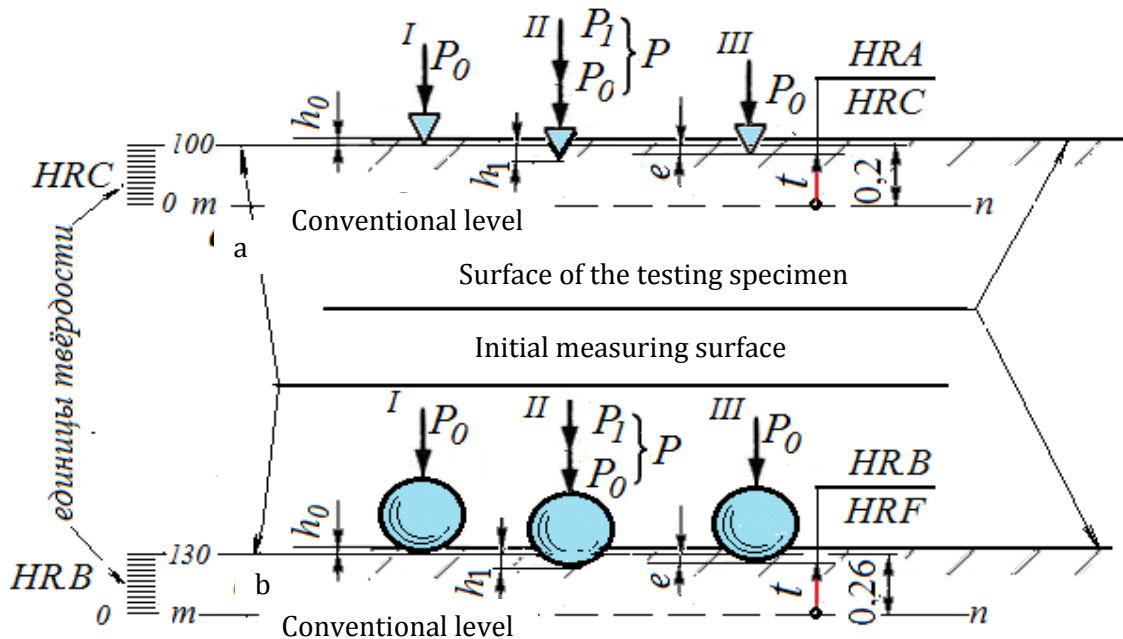

Fig. 1.6. Circuits of introduction of diamond (a) and spherical (b) indenters during Rockwell hardness gauging: I - a preliminary stressing; II - a final stressing; III - gauging

It is considered that Rockwell hardness is the distance *t* in standard units on which indenter at gauging has not reached conventional level. Conventional level *mn* is located, depending on the indenter shape, on distance equal 0,2 or 0,26 mm from an initial measuring surface (fig. 1.6).

All displacements of indenter at hardness gauging are fixed by the hour type indicator on which circular scale define a hardness value (fig. 1.7). The indicator has two scales - black and red. On a black scale there is measured hardness of firm materials in HRA and HRC units, and on red – hardness of soft materials is measured in HRB and HRF units.

Scale C, cone, 150 kgf
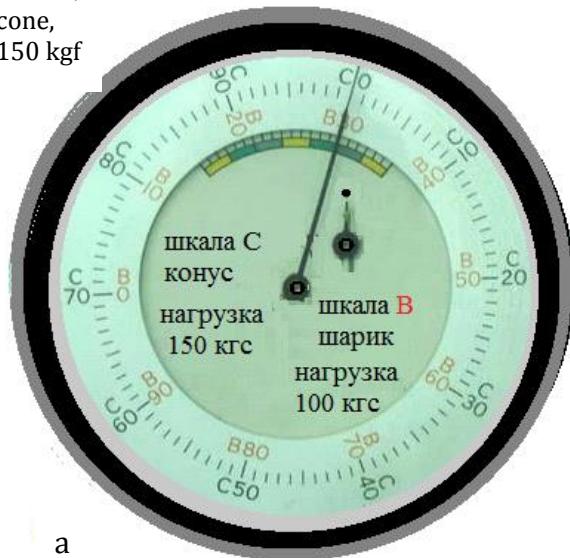
Scale B, ball, 100 kgf
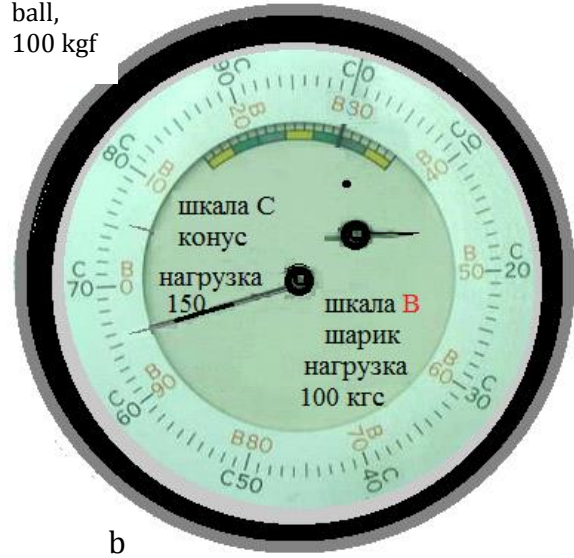
Fig. 1.7. The indicator of a Rockwell press **ТК-2** with clock-face hardness scale: *a* - position of arrows after a preliminary stressing when the small arrow coincides with a point, and big - with scale zero; b – position of the arrows after material testing

The scales graduating marks is identical and correspond to a hundredth part of a scale circle and is equal 0,002 mm of depth of indenter impression. Thus the black scale will have at testing with a conic tip 0,2/0,002=100 divisions, and at testing by a spherical tip (a red scale) - 0,26/0,002 = 130 divisions.

Hardness number is defined under formulas:
• on black scale HRC (HRA) = t = 100 - e/0,002;
• on red scale HRB (HRF) = t = 130 - e/0,002.

Rockwell hardness unit is dimensionless magnitude counting in standard units and also corresponds to indenter axial displacement at 0,002 mm. On a scale C there is usually measured hardness of tempered steels at load P = 150 kgf. On a scale A spend gauging of thin parts or layers. This scale completely coincides with a scale C, but gaugings are carried out at smaller load P = 60 kgf and smaller depth of permeation. At gauging by a steel ball on a scale B (a load of 100 kgf) hardness gauging is carried out at its moderate values (for example, steels after annealing), and on scale F (a load of 60 kgf) hardness of nonferrous metals and alloys is defined. A choice of a load and indentor type for a material hardness test via Rockwell is made according to actual standards. The distance between two prints centers should be not less

than 3 mm.

Virtues of a method of hardness gauging via Rockwell are: 1) possibility of hardness gauging in a wide range both very firm and rather soft materials; 2) prints from a cone and a ball are so small that it is possible to carry out testing on already finished details; 3) testing occupies not enough time, and hardness number is read immediately on a device scale.

Rockwell hardness values can be counted on re-counted to Brinell by means of empirical conversion tables.

3.3. Impact resistance definition [GOST 9454 - 84]

Important characteristic of material reliability is the ductility. *The ductility* is ability of a material to collapse resistance at dynamic loads. The materials demanding the big amount of energy to a breaking (collapse) are called *ductile*. Ductility of a material can be fixed via test data of impact bending or by the form of a breaking.

The test method on impact bending is grounded on collapse of notched test specimens which serves as the concentrator of stresses. Samples can have concentrators of three aspects: an incision in the form of U character with radius of concentrator $R=1$ mm; an acute incision in the form of the V character with $R=0,25$ mm and with fracture T (fig. 1.8). Samples with U-shaped concentrators (samples of Sharpi) apply at an acceptance inspection of metals. At testing of metals of responsible assignment it is recommended to apply specimens of Mesnager with a V-shape incision (details of flying machines, vehicles, capacities of a high pressure and etc.). Samples with T flaw are intended for especially responsible constructions and details where resistance to flaw development has a primary value. For testing the ends of the sample fix horizontally on supports of pendulum impact testing machine with incision side directed opposite to pendulum blade impact. Before testing the pendulum of the impact machine is lifted up to the home position to height H (fig. 1.9) and keep it in this position by a trigger. The floating lever liberated from a trigger, drops down, destroys the sample and due to inertia rises to height h. On a scale of pendulum impact machine the total work of impact K, spent for specimen destruction, is defined or is calculated under the formula

$$K = P\,(H - h) = mgl\,(\cos\beta - \cos\alpha),\ \text{J (kgf·m)}. \tag{1.8}$$

Then this work is divided onto a cross-section of the sample in a place of incision F_0 (use standard bars with $F_0=0,8$ cm^2 is more often).
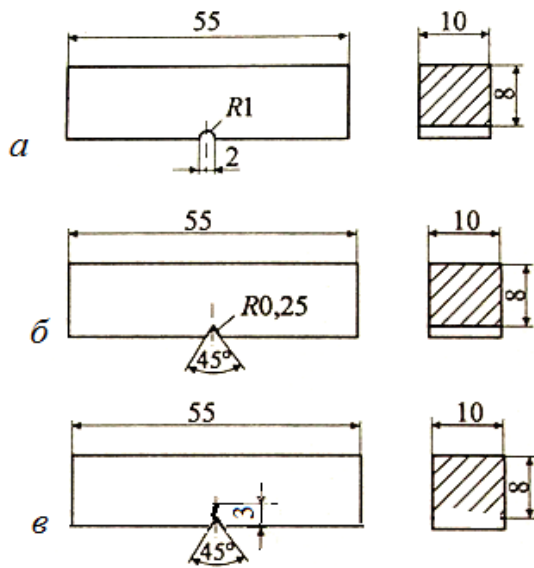

Fig. 1.8. The basic types of ductility test specimens with a various aspect of incisions:
a - **U**-shaped; b - **V**-shaped;
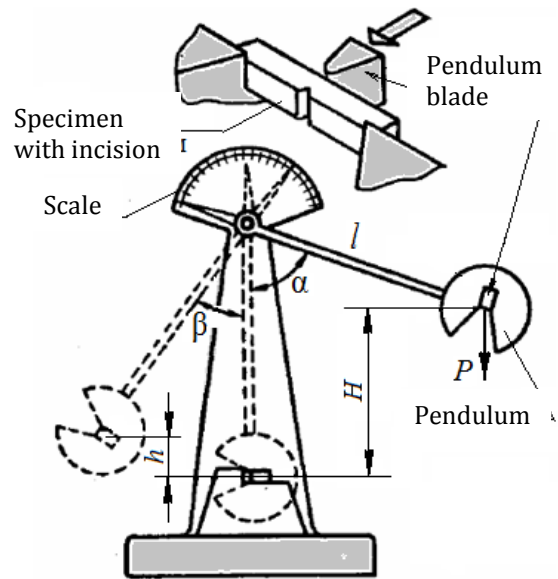c – **T**-shaped with the initiated flaw

Fig. 1.9. The impact test circuit on pendulum testing machine MК-30:
α - a lead angle; β - a flight angle; H and h – lead height and flight height of floating lever accordingly; l - length of a floating lever

Obtained value $KC = K/F_0$, J/cm^2 (kgf×m/cm^2) is called as specific work of collapse or *impact resistance*. The impact strength of a material is more, the ability to collapse resist under the influence of impact loads is better. Total magnitude of work of collapse develops of two components: work for flaw origin A_o and work for flaw spreading A_s, i.e. $K=A_o+A_s$.

Reliability of a material is defined by flaw spreading work. The more A_s, the is less possibility of sudden fragile collapse. For definition of A_s the samples with a fatigue flaw (concentrator) which create on the special vibrator is used. At collapse of such sample the work is expended only on flaw spreading. After a impact test and definition of A_s it is necessary to analyze a fracture aspect. At good material with high enough value of A_s the fracture should be ductile with filamentary or dimple aspect.

Depending on the shape of an incision the impact strength is meant

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KCU, KCV or КСТ. If first two KC characters mean an impact strength symbol, the third character - a concentrator aspect. Then after characters the following first digit displays the maximum energy of floating lever impact (J), the second - depth of the concentrator and the third - width of the sample (for example, *КСТ 150/3/7,5*).

Digits in an impact strength designation are not presented, if at definition of impact work were used the impactor with the maximum energy of floating lever blow 30 J, and samples with the sizes 10x10 mm, with depth of the concentrator of 2 mm for samples with both U and V concentrators and 3 mm - for samples with the T-shaped concentrator.

Deficiency of impact strength as strength characteristic, that it is not used in calculations of details strength. However the practical value of this characteristic is great enough, because it allows evaluating ability of a material to collapse resist.

Having obtained numerical values of mechanical properties as a result of mechanical trials, it is possible to state the technical estimation of strength of metal. As structurally strength material it is possible to consider only such material which in exploitation conditions possesses an optimum combination of strength, plasticity and ductility.

4. A practical part

4.1. Security measures at operation execution

Before to start execution of laboratory work, it is necessary:

1) to pass instructing of safety engineering (SE) and fire safety (FS) and to undersign the control sheet;

2) only those students who have taken place examination on FS and have putted their signature in control sheet of student instructing registration are supposed to execution of laboratory operation;

3) laboratory operation is executed in educational laboratory where electrogears and office equipment corresponding to requirements of SE are placed.

4.2. The equipment and materials

• slide gauge;
• samples from a steel 10, steel 45 and steel У8 for hardness test;
• samples for tensile test (samples for tension of various aspects

before and after a break);
- hardness meter of Brinell ТШ-2М;
- hardness meter Rockwell ТК-2;
- educational movies about test methods of materials on tensile and impact strength.

4.3. Sequence of execution of operation

1. To familiarize with standard samples on a tension and impact strength. To look at videoclips, paying attention to the construction, mode of functioning of testing machines, a technique of carrying out of trials, definitions of strength and plastic characteristics, impact strength of materials.

2. To define relative elongation δ and a contraction ratio ψ (at $l_0 =$ 25 mm) via the samples exposed a tension.

3. To study by means of the educational master (teacher) the construction of used hardness meters.

4. To carry out hardness gaugings via three samples of annealed and tempered steel with a various carbon content by means of Brinell and Rockwell methods, having selected test specifications. Diameter of a print for a method of Brinell is measured in two orthogonally related directions and define as an arithmetical average.

5. To measure hardness of duralumin via scale HRF of a Rockwell hardness meter. At gauging via a method of Rockwell iterate trials on one sample 4 times, accepting an average from three last. For comparison of results it is necessary to translate all obtained data a scale of Brinell HB.

6. To calculate tensile strength based on a hardness value, using the empirical formula for a material being measured.

7. Experimental and calculated data should be putted in tab. 1.1 to make the conclusion about influence of carbon on strength and hardness of a steel.

Table 1.1

Results of hardness tests

Material	Hardness measuring method	Brinell hardness HB	$\sigma_{\text{в}}$, MPa
Low-carbon steel (10)			
Medium-carbon steel (45)			
High-carbon steel (У8)			
Tempered steel (45)			
Duralumin			

8. By results of trials to draw graphs of dependence of hardness and strength of a steel in annealed condition from carbon contents in percents.

9. To make the written record of lab.

Criteria of productivity of operation

Laboratory operation is considered as being executed in the event that the student:

1) has learnt to analyze correctly experimental data of definition of the basic mechanical properties of materials;

2) results of the performed operation are presented accordingly with requirements to record drawing up;

3) has correctly answered all control questions.

Requirements to the record contents and drawing up

The record is made with use of an electronic variant of the description of this laboratory work by carrying over of fragments of the text and necessary drawings to the text of the record and the further editing. This part of the record is made out before occupations at preparation for laboratory researches. On laboratory work the record is made out definitively by importation of calculations and by writing of summaries. The record should have volume no more than 3 … 4 pages. After definitive drawing up of the record the student protects lab to the teacher by the answer of control questions.

Control questions

1. What mechanical characteristics can be defined by trial of materials on a tension?

2. What samples are applied to tensile test?

3. What dependence is figured by the primary tension chart, recorded by a testing machine on a tension?

4. What typical charts meet in practice when tension of metals is?

5. How due to the tension chart to define σ_B and σ_T?

6. What is the variance between a physical and conditional yield point?

7. How to define plasticity characteristics δ and ψ?

8. What is called as hardness?

9. What is the essence of hardness gauging via Brinell?

10. What values of diameters are used for spherical indenters at hardness test via Brinell and what conditions they are selected from?

11. What gear is used for gauging of print diameter at hardness test via Brinell and how to realize the gauging?

12. How Brinell hardness is designated?

13. What are dependences between hardness and strength of a material on a tension exist?

14. What is Rockwell hardness gauging consists of?

15. What types of tips and loads are applied at trial of materials onto hardness via method of Rockwell?

16. In what cases scales *A*, *C* at hardness test via Rockwell are used? In what cases scales *B*, *F?* How to adjust a gear on these scales?

17. How the Rockwell hardness measured on scales *A*, *B*, *C* and *F* is designated?

18. Why smaller depth of permeation of a tip corresponds to higher hardness of a material at hardness gauging via Rockwell?

19. What conditions can be observed at Rockwell hardness gauging (minimum distances between prints centers and from edge of samples, minimum width of samples)?

20. On what scales (red or black) hardness reference at impression of a diamond tip and a steel ball accordingly is performed?

21. What is the method of definition of impact strength consists of?

22. What property of structural materials is called as ductility and what materials are named ductile?

23. What are methods due to it is possible to fix ductility of a

material?

24. What aspects of incisions are used in test specimens on impact strength and what is its role?

25. How the total work expended on collapse of the sample is defined and what two parts it consists of?

26. What is variance between the total work expended on collapse and specific work?

27. Whether is there a variance between specific work of collapse and impact strength?

28. How impact strength of materials is designated?

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STRUCTURE OF STEELS AFTER CEMENTATION AND TEMPERING BY HIGH FREQUENCY CURRENTS

Work purpose

- Study structure of the cemented steels.
- Study structure of steels after tempering via currents of high frequency.

Theoretical part

Cementation (carburizing) - the chemical heat treatment (CHT) consists in diffusion saturation of a surface layer of metal (product) by carbon. The **cementation purpose** - obtaining high hardness and wear resistance of a surface layer of products with maintaining high viscosity of their core that is reached by heat treatment after cementation.

Usually cementations is subjected to products from constructional carbonaceous and alloy steels with the low-carbon content (to 0,25%) and it is carried out at temperatures of 900-950 °C and above, i.e. heating of steel is carried out to an austenitic state (above a critical point of A_{c3}) in connection with the increased solubility of carbon in austenite in comparison with ferrite. With temperature increase of cementation and increase in soaking time the depth of the cemented layer increases. However at increase of temperature over 950 °C, together with acceleration of process of steel carbon saturation, there is also a strong growth of grain of austenite that is undesirable. Therefore high-temperature cementation at 980-1050 °C is recommended to be applied for hereditarily fine-grained steels that are not afraid of austenite grain growth when heating.

Depending on aggregate state of the external environment in which workpiece is placed there is distinguished cementation in firm, liquid and gas environments. In the industry generally apply two types of

cementation: cementation by the firm carburizer and more progressive - gas carburizing.

At cementation in the solid carburizing agent, products stack in metal boxes what are filled up with wood coal mixed from 20...25% of $BaCO_3$ (for process acceleration) and 3,5...5% of $CaCO_3$ (for prevention of agglomeration of particles of the carburizer). When heating in the furnace of boxes with products and the carburizer the atomic carbon which diffuses in steel is formed. Duration of such cementation is 5... 24 hours.

For gas carburizing as the carburizer use the natural gas (consisting generally of methane CH_4); the controlled atmospheres is obtained in special generators; and also liquid hydrocarbons (kerosene, benzene, etc.) that drops to the furnaces hermetic working space where they form the active gas phase what is rich with atomic carbon.

Gas carburizing is the main process at mass production, and cementation in the solid carburizing agent is used in small-scale production. Gas carburizing, in comparison with cementation in the solid carburizing agent, has a number of advantages: cementation time is reduced to 3.... 12 hours since there is no low-heat-conducting solid carburizing agent; it is easy to regulate process of gas carburizing (changing composition of gas and a gas rate) and to mechanize it; working conditions are improved; the working area of workshop is reduced; further heat treatment becomes simpler.

At cementation, as well as at other versions CHT, several processes at the same time proceed:

1. Formation in a circumambient (or in separate reactionary volume) of diffusing element in the atomic (ionized) state *(thermal dissociation);*

2. *Adsorption* ("sticking") of atoms (ions) on the surface of metal with formation of chemical bonds between ions of the saturating element and base metal;

3. *Diffusion* of the adsorbed atoms from a surface in depth of processed metal (or product).

Thermodynamic driving force of diffusion is the difference in

concentration of diffusing element.

As a result of course of these processes the chemical composition of a surface layer is changed, formed a gradient of concentration of the saturating element in surface and underlying layers. Concentration of diffusing element decreases from a surface in depth of metal (fig. 1). As a result of it the structure and properties of steel change. After slow cooling in structure of the cemented layer it is possible to distinguish (from a surface to a core) the following zones:
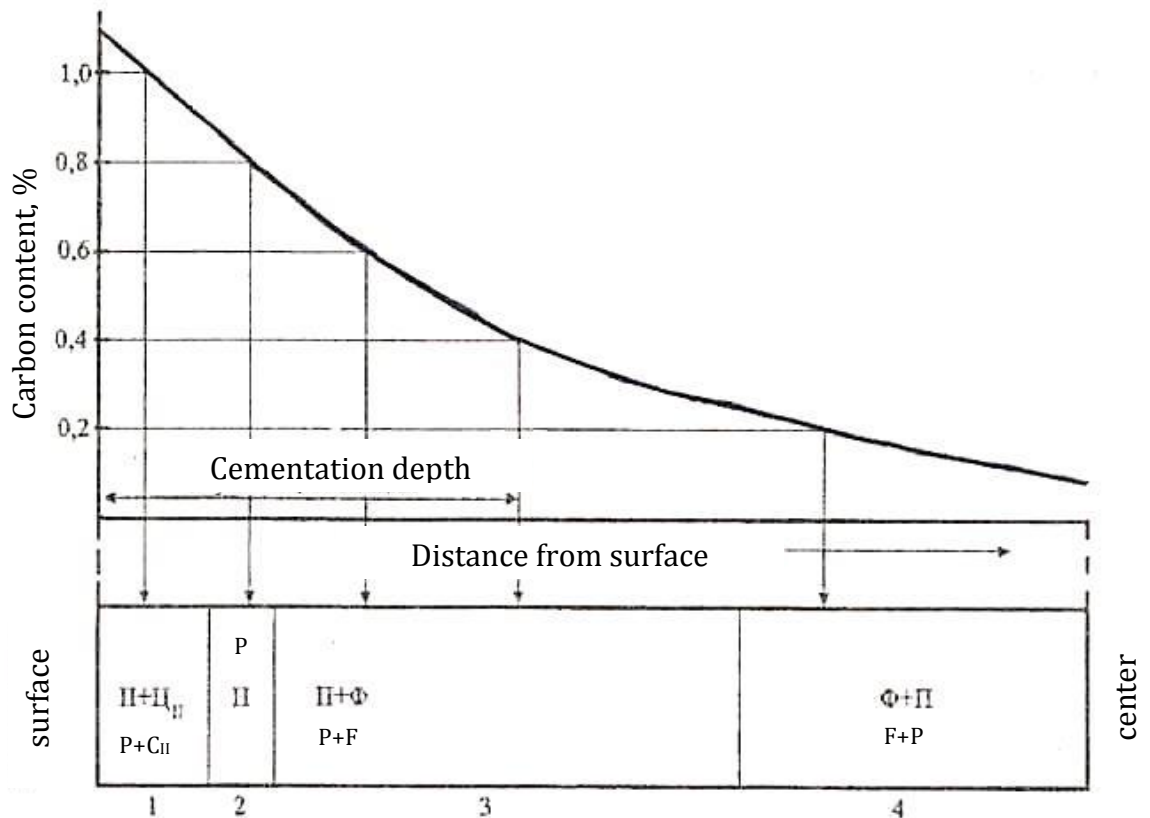

Fig. 1. The nature of distribution of carbon concentration and change of a microstructure on zones in a sample of a low-carbon steel after cementation: 1 - the zone is hypereutectoid, 2 - a zone is eutectoid, 3 - a pro-eutectoid zone, 4 - a pro-eutectoid zone, corresponding to structure of initial steel before cementation.

1) *the hypereutectoid*, consisting of perlite and secondary cement carbide of the austenite forming a grid on the former grain;

2) *the eutectoid,* consisting of one lamellar pearlite, and

3,4) *the pro-eutectoid* zone consisting of perlite and ferrite.

The amount of ferrite continuously increases in this zone as

approaching a core. The core will have structure of initial (before cementation) steel.

Thus, studying structure of cemented steel after slow cooling, it is possible to determine cementation depth. The technical depth of cementation is often defined as distance from a surface to a zone where concentration of carbon is equal to 0,4%, i.e. the structure consists of 50% of perlite and 50% of ferrite. Depth of cementation is determined by metallography (on annealed samples) at magnification 100...500 X. Cementation depth depending on assignment of a product and on composition of steel is usually in limits of 0,5...2,0 mm. Concentration of carbon in a surface layer of a product at the same time is 0,8... 1,0%.

Enrichment of a surface layer with carbon at cementation is intermediate operation because it does not give necessary hardening of a surface. Required hardening of a surface layer of a product is reached by tempering after cementation. Tempering should not only strengthen a surface layer, but also correct the structure of overheating (to crush structure of a core and the cemented layer) arising because of hours-long soaking of steel at a cementation temperature and also to eliminate a cementite grid in the cemented layer.

After tempering of the cemented steel from temperature of 750-780 °C the surface layer acquires structure of martensite with fine particles of globular carbides (secondary cement carbide), and a few quantity of residual austenite. The transition layer containing carbon from 0,8 to 0,6% is also tempered on martensite. The zone with a smaller total carbon (0,6-0,35%) perceives incomplete hardening, i.e. in its structure, except martensite, a few quantity of ferrite as at such total carbon the temperature 750-780 °C are above A_{c1} but below A_{c3} remains. The low-carbon core does not accept tempering and keeps ferritic-perlitic structure with a small amount of perlite ($\approx 15 \div 30\%$).

Final operation of heat treatment after cementation and tempering is the low drawing at a temperature 160 - 180 °C which is reducing residual stresses and not reducing the hardness of surface layers of a product (HRC 58-63)

Chemical heat treatment of steel products, and including

cementation, is rather labor-intensive and long process. For obtaining high hardness and durability in a surface layer of a part in combination with a viscous core in the industry use also surface hardening of medium carbon steel consisting in heating of a surface layer of hardening steel above A_{c3} with the subsequent rapid cooling.

In this case heating for hardening is made by the currents of high frequency (HFC) - the most widespread way, in molten metals or salts, by flame of gas or oxygen acetylene burners, an electric-contact method and also by laser radiation.

Surface hardening with use of induction heating in comparison with CHT, is less labor-consuming, highly productive and in many cases of parts is not of a lower quality than cementation. When tempering by HFC the magnetic field induced in a product leads to emergence of eddy currents and allocation of Joule heat in the heated body. After heating in the inductor (the inductor represents copper tubes with the water circulating inside for cooling of the inductor) the part is cooled by means of the special spray device.

Use of induction heating by currents of high frequency allows to heat a surface of a steel part very quickly. Maximum temperature of a surface is defined by time of transmission of current by the inductor, and depth of heat penetration - current frequency: the frequency is higher, the depth of heat penetration is less (frequency of current can be ranged from 50 Hz to 10^5... 10^6 Hz).

Schematically the schedule of change of temperature on depth of a part is shown in fig. 2.
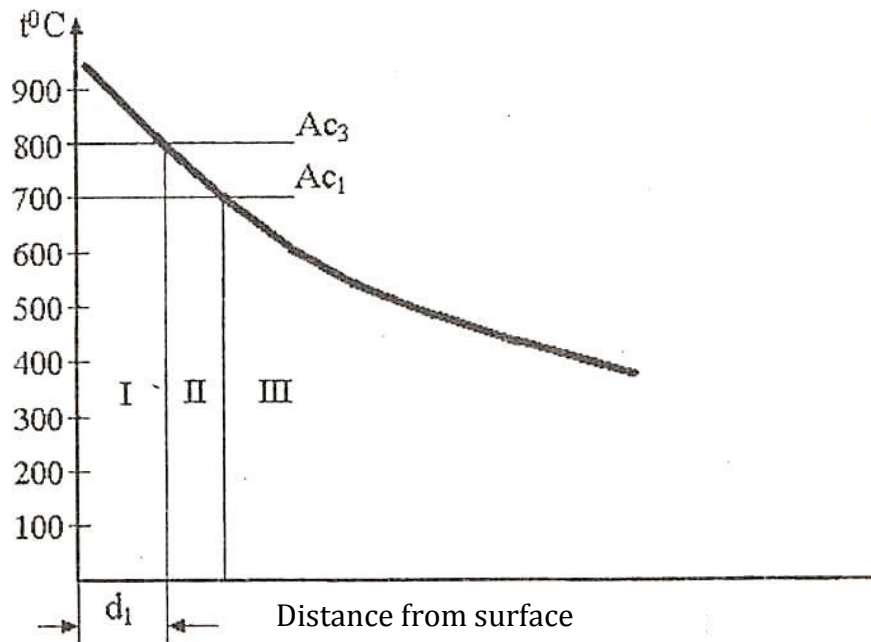
Fig. 2. The scheme of change of temperature on
part depth when heating by HFC

If we heat, for example, steel 45, then at a depth d_1 the temperature will exceed A_{c3}; under a surface layer I there is a second layer heated up to one temperature above A_{c1}, but below A_{c3}. At last, under the second layer, there is a layer III - a core in which reheat temperature has not reached A_{c1}.

At the subsequent rapid cooling the first layer will receive full hardening on martensite. The second layer - incomplete hardening. In structure of this layer except martensite there will be a ferrite which amount is more than the zone is farther from a surface. The third zone - will not apprehend tempering and will keep structure of initial annealed steel, i.e. F+P.

Thus, after tempering the part surface with martensitic structure will have the highest hardness. In process of distance from a surface the hardness decreases.

High hardness of martensite is caused by the distortions of a crystal lattice connected with big internal stresses; high density of dislocations, different borders and subborders complicating the movement of dislocations and also emergence of a phase peening due to increase in

volume at austenite transformation in martensite.

1. Necessary equipment and materials

1. Metallographical microscopes.

2. Collections of microsections

a)   polished section №1 - steel 20 after cementation and slow cooling

b)   polished section №2 - steel 20 after cementation and water hardening from temperature of 760...780 °C.

c)   polished section №3 - steel 45 after tempering of HFC.

2. Order of laboratory work carrying out and record of experiment results

In this work it is necessary to investigate change of a microstructure of samples of steel 20 after cementation, cementation and tempering and steel 45 after tempering of HFC.

For this purpose should place the studied sample on an objective table of a microscope so that under a lens there was an edge of a sample. Slowly moving a microscope table, to study change of a microstructure on sample depth. To put results of observations in a workbook, consistently representing schemes of structures, from a surface to the center zones with taking into account change of chemical composition of samples No. 1 and 2 during cementation and taking into account a gradient of temperatures in a sample No. 3 before tempering. To sketch schemes of structures with pencil in rectangles of 30 x 120 mm in size with allocation of 4 characteristic zones in samples No. 1 and 2 in each of 3 zones in a sample No. 3.

3. Requirements to the report

The report on laboratory work should contain:

1. Subject and purpose of work.

2. Basic theoretical provisions.

3. Drawings.

4. Schemes of structures by samples and zones.

5. Conclusions with an explanation of experimental results according to the work purpose.

6. Answers to control questions.

6. Control questions

1. What kind of chemical heat treatment is called cementation?
2. What is the purpose of cementation?
3. What steels are subjected to cementation?
4. What temperatures the cementation is carried out at and why?
5. To call kinds of cementation depending on aggregate state of the external environment for cementation.
6. List the advantage of gas carburizing.
7. What is the main processes proceeding at cementation?
8. What is taken for the technical depth of cementation?
9. Why after cementation the heat treatment is necessary?
10. What is the tempering of HFC?
11. How the frequency of current and time of current transmission influence on results of HFC tempering?
12. What steel can be subjected to HFC tempering?
13. Whether structures in the center of samples No. 1, 2 and 3 will be different from each other?

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