

PROBLEMS OF THE METALLURGICAL QUALITY
OF STEEL (NONMETALLIC INCLUSIONS)

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The "metallurgical" component of quality is associated not with the nominal composition and heat treatment of the steel, but the melting and forming procedures.

Large changes in melting and casting procedures, wide use of treatments outside the furnace and remelting, and new trends in continuous rolling have had such strong effects on quality that metallurgists have been faced with the following questions: What specific level of metallurgical quality is economically optimal for a given class of steels? What combination of tests should be conducted to guarantee the quality? What service life and what tests to guarantee the quality of steel are economically justified? Metal scientists have not been prepared for the specific problems of quantitative calculations, standardization, and control of metallurgical quality, but modern developments in physics, methods of observation, and integral quantitative description of the structure — from the atomic scale to the macrostructure of the ingot — now permit these questions to be answered.

Only one factor in the metallurgical quality of steel (but the most important) is considered here — nonmetallic inclusions (NI). Scales by which steels can be graded by type and quantity of nonmetallic inclusions (GOST 1778-70) are based on test results over a period of many years. However, methods of grading steels in terms of NI must be perfected on the basis of modern methods of analyzing the structure [1], since it is not known what grade should be specified for a given scheme of selecting the sample and field of view, what volume of testing is economically justified, what is more effective in each specific case — visual or mechanical methods of grading, what is the optimal ratio of final and intermediate testing (for a specific grade of steel and specific application), and when the product should be accepted, rejected, or reinspected for NI.

The most important problem is the mechanism of the effect of NI on the final properties and structure of steel. Metallurgists recognize several methods of influencing the quantity, nature, size, shape, and distribution of inclusions. These are primarily melting, oxidation, casting, and remelting procedures. In some cases modern physics is already capable of answering the question of how inclusions, under what conditions, and in what quantities will affect a given property of steel.

Nonmetallic inclusions can be distinguished from all other structural components not so much by their nature as by the very large range of sizes (10^{-6} –1 cm) in small quantities. With all melting procedures the volume percentage of NI is $c = 10^{-4}$ – 10^{-3} (i.e., ~ 0.01 – 0.1% by volume).

The quantity of NI per unit volume of the ingot determined by direct measurements varies with the particle size d by the rule $n_i = e^{-d/d_0}$ [2], rising from 10^3 to 10^{11} cm^{-3} as the particle size decreases from $d = 40$ μm to $d = 0.05$ μm . However, the volume percentage of inclusions of a given size $c_i = n_i d_i^3$ changes negligibly. For this reason, the basic volume percentage of NI must consist of large inclusions observed metallographically despite the gigantic number of fine inclusions (88% by volume with $d > 1$ μm and only 2% with $d < 0.2$ μm [2]). A more graphic characteristic than NI is the average distance λ_i between NI of given size d_i . For evenly distributed equiaxed nonmetallic inclusions $\lambda_i = d_i / \sqrt[3]{c_i}$, i.e., is 10–20 times larger than the cross section of the inclusions (λ_i is still smaller for nonequiaxed NI in the form of whiskers and films).*

With volume percentage $c \leq 10^{-3}$ of particles of any size there is no arrangement capable of increasing the strength notably, although the particles may play a decisive role in the

*In the plane of the microsection these distances are $c^{-1/6} = 3$ – 5 times larger.

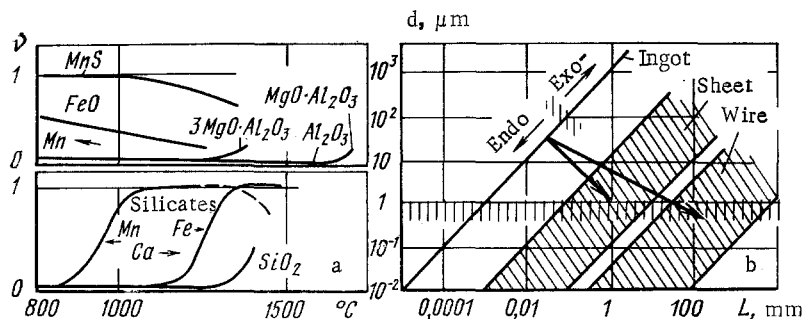


Fig. 1. Deformability and change in the dimensions of nonmetallic inclusions. a) Temperature dependence of deformability [2] (γ is the percentage deformation of the inclusion in relation to the total deformation of the metal); b) change in length L and thickness d of the deformed inclusion ($\gamma=1$) during rolling of sheets and drawing (the vertical hatching indicates the upper limit of inclusions changed by heating of the solid metal).

process of fracture. It is most important that different kinds of fracture are due to inclusions of different sizes.

The principal difference between brittle and ductile fractures is autocatalytic propagation of the crack from a single source. The presence of NI in brittle fracture is accidental. NI are dangerous only as primary centers. At stresses $\sigma < \sigma_T$ the NI splitting or separating from the matrix become the centers of cracks if the critical stress intensity $K_{Ic} < \sigma \sqrt{2d/\pi}$. Thus, only NI with sizes $d > d_{cr} = (\pi/2)(K_{Ic}/\sigma_T)^2$ are dangerous as centers of brittle fracture. For low-carbon steels ($\sigma_T \approx 30 \text{ kgf/mm}^2$, $K_{Ic} > 300 \text{ kgf/mm}^{3/2}$) $d_{cr} > 100 \text{ mm}$, i.e., fracture begins from macroscopic stress concentrators at the surface. Only in high-strength steels ($\sigma_{0.2} \approx 170 \text{ kgf/mm}^2$, $K_{Ic} \approx 200 \text{ kgf/mm}^{3/2}$) is $d_{cr} = 2 \text{ mm}$ and equiaxed NI may be centers of brittle fracture. Inclusions of this type are always accidental [3] and their formation depends not so much on the melting and pouring procedures as on technological discipline; they can be eliminated completely only by remelting.

Determining the concentration of large NI is difficult, although they are visible with the naked eye. On the average, $N_i = c_i F/d_i^2$ inclusions are found in area F . Generally, the total area of the section from one heat is $F = 24 \text{ cm}^2$. With $d_i = 1 \text{ mm}$ and $c_i = 1 \cdot 10^{-4}$, $N = 0.24$, i.e., no inclusions are observed in three cases out of four. As can be seen, the range of observed NI is limited not only by the resolving power of the microscope but also by the accessible area of examination, and only nondestructive testing for accidental NI is possible (even use of ultrasonic defectoscopy reveals particles with $d \geq 2 \text{ mm}$).

If brittle fracture is a danger only for steel in the high-strength condition with large accidental inclusions or films, ductile fracture is practically always controlled by inclusions. Most ductile fractures of steels with a structure of ferrite, bainite, or martensite have a dimpled relief. At the bottom of dimples with cross sections $L = \lambda = 0.5\text{--}5 \text{ }\mu\text{m}$ there are generally inclusions (the possibility of dimples in the absence of inclusions has not been demonstrated). Ductile fracture is not associated with inclusions only in the case of some biphasic structures (in divorced pearlite, for example, dimples are associated with cementite particles). In order for each dimple to contain one NI the average size should not exceed $d = \lambda \sqrt{C}$, i.e., $d = 0.05\text{--}0.5 \text{ }\mu\text{m}$. Ductile fracture begins from larger particles of NI ($4\text{--}16 \text{ }\mu\text{m}$ in cast steel [14]), but finer NI (up to $0.05 \text{ }\mu\text{m}$) occur when microcracks combine into the main crack. In this case the large NI determine the stage of initiation of ductile fracture in the neck and the fine NI contribute to the work of crack propagation.

The effect of NI on the anisotropy of the toughness of rolled products is generally very large. The extent of deformation of the inclusion constitutes some percentage v of the deformation of the metal, depending on the composition of the inclusion and deformation temperature (Fig. 1a). With $v=1$ the deformation of NI is the same as for the base metal and then deformation of an ingot with cross section $B \times B$ to a bar with $b \times b$ reduces

the transverse dimension of inclusions by $k = B/b$ times, drawing them into whiskers $k^2 d$ in length. During preparation of sheets from a slab a k -th change in thickness leads to a change in the thickness of inclusions d/k and in length kd with almost no change in width d . Corresponding changes occur in the distance between particles λ . Generally, for hot rolling $k^2 = 20-50$ and ductile inclusions shift to another range of sizes (Fig. 1b), and therefore the number of metallographically observable inclusions also changes (with drawing $>40-60$ the metal generally becomes "free of inclusions" [5]) and also the mechanism of their interaction. With elongation along the fiber the effective dimensions of the inclusions decrease and breaking of NI slightly changes the plasticity — the relative reduction in section ψ and elongation δ . With elongation across the fiber the effective dimensions of the inclusions increase and the distance between them decreases. For this reason, the NI in the neck of transverse samples separate with smaller deformations and stresses, and they absorb less energy when they join together. Thus, with the same volume percentage of NI with $d \geq 5 \mu\text{m}$ an increase of deformability from $\nu = 1/4$ to $\nu = 3/4$ in steel 35KhN3MFA ($\sigma_{0.2} = 100 \text{ kgf/mm}^2$) at a forging reduction ration of 20 increases the coefficient of anisotropy of the relative reduction in section and the fracture toughness from 1.6 to 3.7 with no changes in the properties in the longitudinal direction [6]. The stretching of sulfides and silicates is still larger during rolling, as the result of which the type of fracture also changes: Even in longitudinal samples it begins with "splitting along" — with separation of lenticular inclusions ("woodiness"). No dimples are observed on transverse samples in this case — the lenticular bridges between NI whiskers are cut, creating a slaty fracture [7, 8]. Early lamination from a film of silicates (in structural steel [9]) or MnS (in rail steel [10]) reduces the overall plasticity. The results of testing sheets "in the third direction" — along the thickness — indicate not only substantial loss of plasticity but also lower strength. The transformation of globules into whiskers reduces the fracture toughness of longitudinal samples of steel 18GTB by one-third and transverse samples by two-thirds [8]. For the same reasons, pinnate inclusions in the ingot (MnS of type II, for example [11]) and especially films are more dangerous than globular NI with the same volume and deformability, since with equal reduction they form a wider band of inclusions. Also dangerous are dendrites and clusters of NI, since any unevenness in the distribution of NI reduces the energy absorbed in ductile fracture.

Although reduction of NI reduces the transverse plasticity, the lamination of longitudinal samples during brittle fracture may also increase the fracture toughness, arresting the main crack. In those cases with unchanged relative elongation and reduction the bending angle of bands and the fracture toughness increase. The practical benefit of macrolamination during fracture depends on the operating conditions of the structure, but fracture toughness tests rarely reflect these conditions.

Undeformed NI may be broken up, forming stringers. The length (and "spacing") of stringers depends on the strength of the particles. In extreme cases a "rare" stringer may have the same effect on the toughness as an isolated "dense" inclusion, but there is presently no criterion of "density" in the mechanics of materials that would make it possible to determine the danger of stringers even though the need is obvious.

In the process of fatigue failure the crack propagates along a wide front and with fracture above the ductile-brittle transition temperature the step per cycle does not generally exceed $1 \mu\text{m}$. For this reason, equiaxed NI have practically no effect on the propagation of the fatigue crack: The number observed in the fracture is no larger than on the microsection. However, NI near the surface of parts, as concentrators of stresses and deformation, may be the centers of fatigue cracks if the sizes of the NI are larger than the sizes of the microroughnesses on the surface from machining, but with contact fatigue if the highest tangential stresses occur at a greater depth than the surface roughness. Therefore, NI have a decisive effect on contact fatigue (crumbling) of smooth rolling surfaces — rails, ball bearings, carburized layers on gears, for example (after carburizing, low-carbon steels are very close to steel ShKh15 in terms of structure, properties, and operating characteristics).

A fatigue crack is nucleated as the result of repeated alternating microplastic deformation* in the overload zone, the width of which is proportional to the cross section of

*In low-cycle fatigue (failure after 10^3-10^4 cycles) macroplastic deformation occurs in each cycle and the NI have the same effect as during ductile "static" failure [4].

the NI. For each stress there is a minimal area required for generation of dislocations and formation of slip bands. For this reason, the first center of fatigue is a very wide zone — from the largest NI.

The thickness of the layer of contact fatigue below the surface of a part is commensurate with the cross section of the inclusions d_i . In this case $N_i \cong c_i/d_i$ of inclusions with a size d_i , although the volume percentage c_i decreases with increasing d_i more rapidly than d_i^{-2} , and therefore there is some "moderate" size of d_i most often encountered in the focus of fatigue. Extremely large (and thus rare) inclusions may cause a sharp reduction of the fatigue limit of individual parts but have a negligible effect on the average value. As the result, after 10^7 cycles 90% of the internal fatigue cracks in rails, e.g., start from NI with a size $>30 \mu\text{m}$. The probability of nucleation of a fatigue crack from a given NI increases from 0.15 to 0.9 as d increases from 0.03–0.12 mm to 0.3–0.4 mm. However, it is most common for fatigue cracks to begin from NI with $d = 0.1\text{--}0.3 \text{ mm}$, since there are more NI of this size [12]. In fatigue tests of ball bearing steel oxides with a size of 6–10 μm prove to be 16 times more dangerous than larger inclusions [13] that rarely penetrate into the layer below the surface. Oxides with a size of 3–10 μm were the source of fatigue cracks in patented wire ($\sigma_b = 183 \text{ kgf/mm}^2$) [14].

Residual microstresses of thermal origin around NI are not dangerous, since microplastic deformation during fatigue eliminates them before the nucleation of cracks. The smaller the modulus of elasticity of inclusions, the higher the stress concentration during elastic deformation. In order of decreasing modulus of elasticity, inclusions can be arranged as follows: TiN, Al_2O_3 , SiO_2 , (Fe, Mn)S, CaO [13]. On the contrary, the concentration of deformation during plastic flow measured by the photoelastic method decreases by a factor of 1.5 from Al_2O_3 to SiO_2 to MnS [12]. The unevenness of flow (and thus plastic concentration of cyclic stresses) is largest around rigid inclusions where triaxial stresses split the NI or separate it from the metal [15]. In addition to rigidity, the faceting of the NI is also important: At inclusions with absolutely sharp edges the theoretical stress concentration is 50% higher than at spherical inclusions [16]. Rigid acute-angled inclusions of corundum in steel ShKh15 are more dangerous than globular silicates or sulfides. A sulfide film enveloping corundum wets it, covering the edges with a "cushion" of low rigidity. For this reason, the contact fatigue depends on the ratio of the concentration of oxygen and sulfur in the steel.

Contact fatigue is associated also with breaking up of oxides during pressing. The "burnishing" of plastic NI may affect the fatigue of structural steels in two ways — on the surface of lenticular NI the fatigue crack runs in a single jump, due to which the average velocity of the crack increases, but branching of the crack along the NI postpones the moment of fast fracture. Thus, some optimal degree of lamination of the structure is possible in terms of fatigue — for spring steels, for example.

Nonmetallic inclusions also affect all processes of fracture indirectly by way of the grains. When boundary migration induces tension but inclusions inhibit it the grains cease growing, reaching a size $D = 8d/3c$ [17]. Since $c \leq (10^{-3}\text{--}10^{-4})$, while $D = 10\text{--}100 \mu\text{m}$, only NI with sizes $d \leq 0.01\text{--}0.1 \mu\text{m}$ can affect grain growth. Such inclusions always coalesce near the solidus. They must be precipitated at a lower temperature and belong to those phases whose solubility changes substantially at fairly low temperatures. At electron microscopic studies have shown, these are particles of AlN [18] or MnS [17] with sizes of 0.02–0.1 μm at a concentration $n \cong 10^{13} \text{ cm}^{-3}$. The concentration of sulfur or nitrogen in austenite depends on the heating conditions for rolling, and the precipitation of such NI on the subsequent cooling conditions, which also determine the "inherited" size of grains retained after heat treatment (since this temperature is generally below the temperature to which the steel is heated for rolling). Jumplike grain growth ("overheating") occurs when these NI either go into solution or coalesce. For this reason, the susceptibility of steel to overheating can be influenced by alloying and microadditions (changing, e.g., the solubility of sulfur and nitrogen or the excess concentration of aluminum in the solid solution).

Inclusions with a cross section $d \cong 0.01 \mu\text{m}$ are not centers of fracture. However, they become centers of ductile grain-boundary fracture as they grow larger (inclusions of CrS in steel of the 40Kh2N4M type, e.g. [19]), and their fusion in grain-boundary films leads to intergranular fracture. The retention of networks of "coarser" inclusions leads with overheating of alloy steel to "inheritance" of large grains during annealing and the associated

brittleness. The transformation of the system of boundaries into a flat layer with inclusions of AlN during drawing may be the reason for lamination [20].

Control of NI of submicron size during melting (the influence of their components in the solid solution on their composition, solubility, and residual concentration) and during hot working has not been investigated for its influence on the nature of grains in the steel, its hardenability, susceptibility to overheating, and tendency to lamination.

The motive force of the polymorphic $\gamma \rightarrow \alpha$ transformation is two orders higher with moderate supercooling than during grain growth. For this reason, NI affect the decomposition of austenite not by way of the mobility of the α/γ boundary but by way of the original austenite grain size and the concentration heterogeneity of the metal. For example, silicate whiskers are enriched in silicon adjoining a band of metal 1-10 μm wide. Raising the thermodynamic activity of carbon, silicon drives it from the layer around the NI and creates ferritic banding that is not eliminated by heat treatment. Sulfides (Mn, Fe)S, on the contrary, are enriched in manganese from the adjoining metal, which leads to an increase in the change points of the steel. This broadens the range of temperatures and transformation times. Thus, treatment of acid steel 35KhN3MFA with calcium-silicon shifts the range of the bainitic transformation 50° and broadens the transformation time by a factor of 2-5, other conditions being equal [21].

Controlling the quantity, morphology, plasticity, and brittleness of NI by changing the deoxidation procedure, treatment outside the furnace, and casting procedure (including the limitation of secondary oxidation) and matching the type of inclusions determining the properties with rolling or forging temperatures and the proportions of the sections of ingots and semifinished products is still inadequately used to obtain the necessary values of the toughness and anisotropy of the steel.

The question of toughness and nonmetallic inclusions does not at all lead to the highest possible purity of the steel. For an economically valid increase of the toughness it is necessary to determine the upper limits of the sizes, quantity, and elongation of inclusions for each class of steels and the operating conditions that would guarantee the necessary reserve of toughness, weldability, and machinability. It is also necessary to differentiate the characteristics of toughness and plasticity under conditions of failure in structures and ensure statistically reliable methods of determining NI for melting and manufacturing procedures. Today, our information is still inadequate on the variations in the character of inclusions due to permissible variations in techniques of gas elimination, secondary oxidation, heating of ingots, et al. Detailed studies are needed to guarantee a consistent improvement in the properties of steel as the result of improving the "metallurgical quality."

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CRITERIA FOR DETERMINING THE WORKING

CAPACITY OF STRUCTURAL STEELS

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Structural steels are among the basic materials used in many branches of industry and therefore it is very important to establish the criteria for determining the working capacity (reliability)* of structural steels, i.e., determine the resistance to fracture.

The purpose of this work was to establish criteria for determining the structural strength (working capacity) of alloy structural steels with yield strength as high as 100 kgf/mm² and testing methods suitable for wide use that make it possible to establish the quality of the metal with extremal operating conditions of machine parts.

Various low-carbon and medium-carbon alloy structural steels were tested.

After rolling, the steel was subjected to preliminary heat treatment — quenching from a temperature of $A_c + (50-80)^\circ\text{C}$ and tempering at $650-670^\circ$ or tempering alone (without quenching).

The basic final heat treatment is given in Table 1.

In conformity with the T-T-T diagram, 40-60% austenite remains untransformed in the process of quenching to upper and lower bainite (treatments 3-6). During subsequent cooling a large part of the retained austenite transforms to martensite.

In treatments 2, 4, 6, 7, and 8 the tempering time was regulated to obtain the same hardness as after treatment 1. Tempering at $500-525^\circ$ (treatment 7) greatly embrittles the steel and has a slight effect on the hardness after tempering at $650-660^\circ$.

The austenitizing temperature was selected so that ~70% of the carbides formed in the process of preliminary heat treatment were dissolved in austenite. The austenite grain size was grade 7-9 in this case. Austenitizing at $1100-1200^\circ$ induced solution of ~95% of the carbides and a grain size of grade 1-2.

The structural strength of all steels tested differed greatly after heat treatment under these conditions.

Numerous methods of testing to determine the working capacity and also other methods were used in this work.†

*The terms structural strength, reliability, and working capacity used by various investigators are synonymous.

†In particular, dynamic and static bending tests at temperatures from $+150$ to -196° were made with five types of samples with different notches — type I with a Mesnager notch, overall cross section 10×10 mm, notch depth 2 mm, root radius $r = 1$ mm, net section at the notch 8×10 mm; type II samples were the same as type I but the notch had an included angle of 45° , $r = 0.25$ mm; samples of type III were the same as type II but with a fatigue crack 1.0-1.5 mm deep at the bottom of the notch, overall cross section 11×10 mm, net section $(7.5-8) \times 10$ mm; samples of type IV were the same as type II but with three identical notches, two of which were lateral, overall cross section 10×14 mm, net section at the notch 8×10 mm; samples of type V were the same as type I but with $r = 2.5$ mm.