Effect of
Surface-Active Media
on the
DEFORMATION OF METALS

by

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PREFACE

The object of this monograph is to describe researches into the effect of surface-active media on deformation processes in metals. Most of the work described was carried out over the past ten to fifteen years at the Department of Disperse Systems in the Institut Fizicheskoi Khimii, Akademii Nauk SSSR (SSSR Academy of Sciences Institute of Physical Chemistry).

These researches led to the discovery of a number of new effects, which form the result of the interaction - chiefly by adsorption - between a deformed metal and the surrounding medium containing a surface-active agent. They include a lowering of the yield point of metal single crystals, peculiar modifications in the surface structure of metal, an increase in creep rate, a reduction of fatigue strength, and a deformation-assisting electrocapillary effect.

The part dealing with the effect of a medium on the fatigue strength is based on researches by G. V. Karpenko's group carried out at the Institut Stroitel'noi Mekhaniki Kiev (Constructional Mechanics Institute), and later on also at the Institut Mashinovedeniya i Avtomatiki, Lvov, (Institute of Mechanical Engineering and Automation), under the Ukrainian SSR Academy of Sciences in close collaboration with the SSSR Academy of Sciences Institute of Physical Chemistry.

This collaboration between the Institutes of the All-Union and of the Ukrainian Academies proved exceedingly fruitful. It produced valuable results, which not only pointed to new directions for the further development of this new scientific field but also showed the possibility of various industrial applications.

The development of the new branch of physico-chemical research in this country aroused a growing interest in related problems abroad. Andrade's group in Cambridge, whilst experimentally confirming an increase of creep rate due to adsorption, interpreted it in terms of a disruption of the oxide layer normally present on a metal surface by the surface-active agent. This interpretation was refuted by our own researches, which proved that the principal effects are attributable to the interaction between the metal and its surrounding medium through adsorption. Simultaneously,

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during 1951-1953, extensive experimental investigations by the noted
German metal physicist Masing and co-workers (Goettingen) lead to conclu-
sions confirming those reached by ourselves, and refuting Andrade's.

This new field of research which is concerned with the effect of the
external medium on deformation- and rupture phenomena, and with certain
structural changes in solids, is of considerable interest, as it points to
new methods of controlling the microstructure and fundamental properties
of metals and alloys. Such phenomena are, naturally, of practical impor-
tance during metal working operations and in actual service conditions.
The new field of research may be described as bordering on solid state
physics, metallurgy and the physical chemistry of surface phenomena. Its
many ramifications and interests obviously call for its further extensive
development.

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INTRODUCTION

The effect of the surrounding media on the mechanical properties of solids in particular metals has, hitherto, been mainly discussed in terms of chemical (corrosive) action, such as electrochemical corrosion or dissolution processes.

Soviet physical chemists, however, have found that the effect of a surrounding medium is not limited to chemical interaction with the metal[1]. Adsorption (even reversible) of typical surface-active components from this medium often accelerates deformation and destruction of solids much more effectively than any chemical reaction.

This effect of adsorption in accelerating deformation or reducing the strength of metals can be attributed largely to a lowering of the surface energy of the metal; this assists in the generation of plastic shears and the development of various defects at lower stresses. All subsequent effects are initiated by this primary adsorption action[1].

The phenomenon primarily affects the surface defects. These ultramicroscopic defects or weak spots, originating during the growth of every solid, are as fundamental as the ordered structure of its lattice. During the elastic and plastic deformation of the metal, existing structure defects not only continue to develop but new defects originate in large numbers in consequence of plastic shears along the glide planes. During deformation a real solid behaves as a heterogeneous material consisting of two phases with different elastic-plastic properties; the one an "ideal" medium completely free from defects, the other consisting of defects only. The character of these defects, their topography and distribution in the volume and over the surface of the solid is, as yet, not fully understood. Schematically, the defects may be considered as microscopic or ultramicroscopic cracks of wedge shaped cross-section; the mouths or openings of these microwedges are characterized by a fully developed surface with a

normal value of the specific free surface energy, $\sigma^{(1,2)}$. The free surface energy of the microcracks is determined by their width h,

$$\sigma_h = f(h)$$

and increases from zero at the tip to the maximum normal σ value at the open end. It is known that the presence of such defects in the structure of solids has an important bearing on their mechanical properties; a case in point is the low observed value of the strength compared with the theoretical value calculated from crystal lattice theory.

For example, a rigorous calculation of the tensile strength of rocksalt crystals leads to values in the region of 400 kg/mm², while in actual practice the strength is more like 0.5 kg/mm². Similarly, the theoretically estimated strength of metals is of the order of 10^3–10^4 kg/mm², although their real strength does not exceed a few hundred kg/mm².

The discrepancy can be explained in this way: at the instant of fracture, deformation energy which was previously distributed over the total volume of the deformed solid, is concentrated near the defective region, i.e. around the microcrack. The stress concentration factor at the tip of this crack may raise the externally applied stress close to the theoretical rupture stress.

This is effectively illustrated by the well-known experiments of A. F. Ioffe on the tensile failure in water of rocksalt. The surface layer containing the microcracks was dissolved away, producing in some instances a tensile strength as high as 160 kg/mm², which is close to the theoretical strength.

The part played by structural defects is also apparent in the so-called size effect, that is, in the relationship between the strength and size of test pieces of a given material. Thus, the strength of a 16μ glass fibre is 107 kg/mm², that of an 8μ fibre 207 kg/mm², and the strength of a fibre 2.5μ thick is 560 kg/mm² [3].

Both the structure defects i.e. the "microcracks" present initially in the solid, and those originating during its plastic deformation, reduce its strength and facilitate deformation and failure. They also play an important part in the interaction between the deformed solid and the surrounding medium, providing the path for penetration of surface-active agents into the solid, thus enabling them to influence the kinetics and dynamics of its deformation.

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During the progressive development of surface-microcracks, active
molecules from the surrounding medium enter by so-called two-dimensional
migration - the mobility of adsorbed molecules along the adsorbing surface.
The surface-active molecules tend to cover those newly formed internal sur-
faces to which they have access, by a uniform adsorbed layer. In addition,
the outward face of the adsorbed film may be strongly lyophilic, that is,
it may show molecular affinity to the surrounding liquid, and lyophilize
the freshly developed internal surfaces.

The high solid-liquid wetting energy will produce molecular forces
which bind not only the adsorbed film but also the transient solvate
layers in the liquid. Although the adsorbed films are usually monomole-
cular, the thickness of the solvate layers, according to B. V. Deryagin[4]
is equivalent to some hundreds or even a thousand molecules, and may
even be 0.1μ In such disperse transition layers the molecular bond with
the solid surface becomes progressively weaker and the orientation of
the molecules less marked as the distance from the surface increases.

The force inducing the adsorbed films into the microcracks is the
decrease of surface energy caused by their penetration, which is equivalent
to a two-dimensional pressure

$$\sigma_h(0) - \sigma_h(G) = P_s$$

It is expressed by the force per unit length of linear boundary of the
adsorbed film in dyne/cm-erg/cm^2. In the expression for P_s, $\sigma_h(G)$ is the
specific surface energy at an internal surface covered with adsorbed film
of saturation G, and $\sigma_h(0)$ is the specific surface-free energy of the
metal in a vacuum (or pure solvent). In the regions of cracks accessible
to adsorbing molecules, virtually all σ_h values are close to σ. On the
basis of Gibb's thermodynamic equation for dilute solutions,

$$G = \frac{dP_s}{dC} \times \frac{C}{RT}$$

where C is the equilibrium concentration of the adsorbing material in the
external medium. Dilute adsorbed layers corresponding to the two-
dimensional gaseous state

$$P_s = RTG$$

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that is, the expression for the two-dimensional pressure is analogous to that for the pressure of 1 mol of dilute gas approximating to an ideal gas:

$$P = RTC$$

When the liquid reaches the mouth of the microcrack, the molecules which are most readily adsorbed, rush forward along both surfaces almost to its tip where further progress is arrested by the steric obstruction because of their own size. The critical gap width is equal to one or two diameters of the adsorbing molecules. It is in particular at these regions in the crack that the two-dimensional pressure of the adsorption film is applied to each unit length

$$P_s = \sigma(0) - \sigma(G)$$

acting in the direction of further propagation of the crack, analogous to the two dimensional pressure of a monomolecular layer of organic surface-active materials at the surface of clean water. This pressure by the absorbed layer, or the reduction of free surface energy due to adsorption in conjunction with other factors, is the most important effect contributing towards deformation and failure of the solid.

The mechanism by which adsorbed solvate layers penetrate from the external medium into a growing microcrack is obviously not based on the induction of a liquid of given viscosity into a system of capillaries according to the laws of hydrodynamics. Rather it is a two-dimensional migration of separate surface active molecules along both crack surfaces under the effect of a two-dimensional pressure. This ensures, in accordance with experiment, rapid filling of the microcrack by adsorbed layers, and, by contrast with the induction of a viscous fluid, is independent of the width of the microcrack if the adsorbed molecules are smaller than the gap between the crack walls. Moreover, the spreading of the adsorbed molecules during the two-dimensional migration is independent of the condition of wetting of the solid surface by the surrounding liquid. On the other hand, the adsorption of a liquid medium is determined by wetting when incomplete, and characterized by a definite contact angle θ.

For complete wetting, the capillary pressure is a maximum defined

by:
$$P_m = \frac{2\sigma}{h}12$$

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Complete wetting corresponds to the unlimited spreading of a liquid over a solid surface. Such spreading is a spontaneous isothermal process, i.e. is always associated with a decrease of free energy for each following cm^2 of wetted surface. The decrease

$$- dF/dS = (\sigma_{32} - \sigma_{31}) - \sigma_{12}$$

must, consequently, be positive

$$- dF/dS > 0, (\sigma_{32} - \sigma_{31}) > \sigma_{12},$$

where σ_{32}, σ_{31} and σ_{12} are the values for the surface free energy at the following interfaces: solid 3 - medium 2 (usually air), solid 3 - liquid 1, and liquid 1 - medium 2. If the condition of total spreading is not fulfilled, i.e. if

$$- dF/dS = (\sigma_{32} - \sigma_{31}) - \sigma_{12} < 0$$

or

$$\left| \frac{\sigma_{32} - \sigma_{31}}{\sigma_{12}} \right| < 1$$

then the liquid 1 forms with the solid surface 3 a finite contact angle which is determined by

$$\frac{\sigma_{32} - \sigma_{31}}{\sigma_{12}} = \cos \theta$$

where

$$+ 1 \geqslant \cos \theta \geqslant - 1$$

In this case the capillary pressure, which plays the part of the inducting force, is always smaller than its maximum value $P_m = 2\sigma_{12}/h$, and is

$$P = 2\sigma_{12}/h \times \cos \theta = P_m \times \cos \theta$$

The capillary pressure is inversely proportional to the gap width and can therefore attain very high values which may reach some hundreds or even thousands of atmospheres for the narrowest microcracks. At the same time, because of the high viscous resistance, the induction rates are so small that, in practice, the liquid does not fill such narrow microcracks.

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The free surface energy per cm^2 of a system consisting of a fairly thick $(h > h_m)$ interlayer (film) of a liquid 1 between two flat surfaces of a given solid 3 which it completely wets, is $F = 2\sigma_3$, as the film has two sides. B. V. Deryagin[5] showed experimentally that when two surfaces are brought together slowly (reversible isothermal approach), the intervening film thickness decreases with the free escape of the liquid into the surrounding medium. Once the film attains a thickness h_m equal to double the thickness of the transitional liquid layer at the solid interface, the system free energy will increase continuously due to the energy associated with the decreasing film thickness.

For each cm^2 of film of thickness $h \leqslant h_m$, there is an excess free energy

$$F = 2\sigma_{31} + E(h),$$

for $h \geqslant h_m$, $E(h) = 0$. The excess energy of a thin strong film which rapidly increases with a decreasing film thickness h, can be visualized as the result of some normal "cleavage pressure"

$$P(h) = -dE(h)/dh,$$

always resisting further decrease of film thickness and balancing the external forces. During penetration of liquid into a narrow wedge shaped gap, the excess energy resulting from the cleavage pressure of the wetting film grows at the expense of the wetting energy, which represents the maximum energy. During wetting of a free surface of a solid, the whole wetting energy is liberated as heat; simultaneously, the whole wetted surface is covered by a fairly thick liquid layer. Penetration of a microcrack by liquid in the form of a thin wetting film results in transformation of the "volume liquid" into an unbalanced thin film. The wetting energy of the internal surfaces is not liberated in form of heat, but partly stored as the excess energy of a thin film - the cleavage pressure.

When adsorbed layers forming in microcracks increase the physicochemical affinity of the solid and liquid, i.e. the wetting energy, then either the cleavage pressure will increase for the same thickness of wetting film, or the film thickness will increase for the same cleavage pressure.

As was shown earlier, the internal surfaces of the microcracks may become more lyophilous, i.e. the thicknesses of the liquid solvate layers bound to the surface by molecular forces, may increase. Because of the very small rate of capillary penetration, however, the cleavage pressure

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of such thick layers can hardly intervene to an important extent in the deformation, as the liquid solvate layers cannot penetrate to any appreciable depth during the period of deformation.

The importance of the solvate layers increases however, when the load is removed from the deformed solid, i.e. during the closing up of microcracks under the action of molecular forces. In this case, since cleavage energy is accumulated because of the work done by external forces, its value is only limited by the thermodynamic strength of very thin liquid films, and may attain 10^2-10^3 kg/cm^2.

The cleavage pressure of solvate films, especially in the presence of adsorbed layers, may considerably slow down the closing of microcracks and may even completely prevent it when the molecular forces acting in the narrowest parts of the microcrack are insufficient to squeeze out the limiting thin solvate and adsorbed layers.

This fact assumes special significance in all elastic-kinetic phenomena accompanying the deformation of solids, above all in elastic after-effects and in hysteresis. The solvate layers play a specially important part in cyclic processes, such as fatigue.

In physical mechanics and molecular physics, and even more so in the different departments of mechanical technology, the mechanical properties of solids and their behaviour under different conditions of deformation and failure have hitherto been studied without paying sufficient attention to physico-chemical factors arising from the presence of an external medium. It has been assumed generally that as far as solid bodies of normal dimensions were concerned the mechanical properties at a given temperature and stress are determined solely by the internal nature of the solid.

As will be shown later on, systematic studies of the effect of external media on deformation processes in solids have not only led to the discovery of important new effects and laws, but also to a new appreciation of the mechanism underlying deformation in a number of general cases.

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CONCLUSIONS*

It is evident from the material presented on the preceding pages that the study of the divers aspects of the physico-chemical effects of the surrounding medium on deformation and mechanical failure in metals now forms a new field of scientific research touching molecular physics, physics of solids, and physical and colloid chemistry. Opened up and developed mainly by Soviet scientists, it may be considered broadly as a department of mechanics, relating the mechanical properties of solids, the chemical constitution and structure, on the one hand, and the properties of the surrounding medium in which the deformation and failure processes take place on the other.

During the past few years the scope of these researches has been considerably extended. For example, M. S. Aslanova[91] showed that during prolonged loading in a surface active medium silicate glasses (glass threads between 3 and 60 μ in diameter) which are ideally elastic at room temperature display a distinct elastic after-effect and even creep-type flow, in addition to a considerable loss of strength. These effects were specially evident in aqueous solutions of surface active materials not interacting chemically with the glass surface, and were found to increase with decreasing thread diameter, yet were virtually absent on glass fibres less than 3-2 μ across. The obvious reason would seem to be that the thin threads contain a much smaller concentration of active defects, such as fracture nuclei, in the surface layer which, as is well known, also accounts for the increased strength of thin threads.

A considerable reduction of fatigue strength was also found by Orowan[92] for large diameter glass specimens in an active medium (water) which can only be interpreted by an adsorption-assisted avalanche-like development of surface fracture nuclei if the stress is maintained for a sufficiently long time. Under these conditions the fatigue strength can be halved due to the effect of the medium. This means that a tensile stress half the value of that leading to failure is sufficient to cause failure if applied for a sufficiently long period (several hours or days). The strength of large

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* The author's heading "Conclusion" is rather misleading. (*Translator*).

glass specimens under rapid loading is virtually independent of the presence of an active medium; for sufficiently thin threads some reduction is observed, though this is much smaller than in experiments of long duration.

G. I. Logginov, in the Institut Fizicheskoi Khimii AN SSSR, (Institute of Physical Chemistry) showed[92][93] that mica crystals (foils) when subjected to long-time loading in vacuum or in dry air, behave as virtually elastic solids. Yet in water, especially water containing surface active agents, a slowly increasing (some days) considerable deformation due to elastic after-effect is observed which disappears just as slowly after unloading. It was also shown that under the effect of reversible adsorption of water and electrolytes, elastic gypsum specimens, both in polycrystalline and monocrystalline form, exhibit typical creep even under small loads, leading eventually to failure. This also explains another well-known phenomenon, the development of dangerous creep of gypsum constructional components in a damp atmosphere.

It may be appropriate at this point to note that the existence of considerable adsorption effects in conditions of creep and fatigue strength (with gradual breaking up of the solid structure along the weakest spots progressing from the surface) alone confirms the decisive importance of the penetration of the adsorbed materials into the surface defects.

As shown in IV the English physicist Andrade, based on our results and his own, experimentally observed adsorption-assisted increase in the creep rate of cadmium single crystals (natural acid-etched surface), attempted to attribute all adsorption effects to the assisted breakdown of oxide films in a surface active medium during deformation, having previously shown that metallic flow is slowed down by the presence of oxide films. Further work by V. F. Rozhanskii, Chair of Colloidal Chemistry, MGU[42], and V. S. Ovstrovskii in the Institute of Physical Chemistry[94,95] explained the true influence of oxide films on the value of the adsorption effect. Rozhanskii showed that in the initial stages of extension of a metal single crystal circular ruptures appear in the thinnest oxide films at regions where considerable overstresses develop, i.e. stress concentrations in the surface layer forming a brittle shell. The presence of a thin oxide film sets up a state of stress in the surface layer of the deformed single crystal favourable for the adsorption effect. He showed further that the creep rate in a vacuum is considerably increased by adsorption of surface active vapour on tin single crystals from which the oxide film had been removed completely by reduction in a glow discharge (under very low

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hydrogen pressure). Under these conditions, as shown in IV, the adsorption effect increases with decreasing specimen length and becomes considerable for fairly short specimens, corresponding to a non-uniform stress state. The presence of thin oxide films at the specimen surface, by producing overstresses in the described manner intensifies the adsorption effects in longer specimens. Ostrovskii showed that this action is only obtained in the presence of very thin oxide films: when extending single crystals at a given constant rate specimens with surface oxidation display the effect even at small rates of extension, whilst unoxidized single crystals show the effects only when extended at a rate approximating to the optimum value. With increasing oxide film thickness the adsorption effects become weaker and will in practice cease completely since the screening action of the film prevents access of the surface active agents to the metal surface. Electrocapillary effects on cadmium single crystals and the relationship between fatigue strength and the potential difference at the metal-electrolyte boundary are completely analogous to conventional electrocapillary curves for the surface tension of a molten metal, such as mercury or gallium; this justifies the conclusion that the adsorption effects facilitate the deformation of the metal itself and are a particular characteristic of clean metal surfaces. This view is confirmed by Masing and co-workers[41,43] who obtained similar results on mono- and polycrystalline lead, tin, zinc, cadmium, silver, gold, and platinum in an aqueous electrolyte solution with electrocapillary effect, and in non-polar liquid hydrocarbons containing surface active agents.

Bowden and Tabor[96] established a reduction in the coefficient of friction along the electrocapillary curve under conditions of boundary lubrication between metallic surface (platinum in an aqueous electrolyte solution).This effect is obviously related to the cleavage pressure of the electric double layer which prevents direct contact between the two solid surfaces. It is also obviously connected with the adsorption assisted deformation and, in particular, the deformation or reduction of strength of metals due to the electrocapillary effect studied by E. K. Venstrem in this laboratory, similarly as the reduced friction due to the lubricating action of adsorbed layers between solid surfaces is, to some degree, comparable to the reduction of strength by the adsorption effect. As observed earlier on, the electrocapillary hardness reduction curve was studied by Venstrem during plastic deformation of metals, graphite and some sulphides by the pendulum method[33].

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These researches[97], carried out in close collaboration with Academician A. F. Frumkin and co-workers, led to a new method of determining the "zero point" potential i.e. of the points of zero charge at the metal surface, an important electro-chemical characteristic.

Bockriss and Parry-Jones[98] attempted similar measurements on copper, nickel, cadmium and lead, and also arrived at a relationship analogous to the electrocapillary curve.

Since in Bockriss's experimental conditions the pendulum oscillations were damped by friction between solid surfaces rather than by deformation, i.e. at fairly small stresses in the surface layer of the metal, the electrocapillary effects must be attributed to the effect of the medium on sliding friction rather than on deformation or breakup of solid surfaces. The pendulum characteristic is then the relative rate of decrease in the damping of the pendulum amplitude $-\frac{1}{\alpha} \times \frac{d\alpha}{d\tau}$, which is the reciprocal of our "pendulum hardness":

$$H = - \frac{1}{\frac{1}{\alpha} \times \frac{d\alpha}{d\tau}}$$

Obviously with higher pendulum loadings, or when using a sharper knife-edge bearing with smaller contact surface such as the rough glass sphere used by Venstrem, the damping will be mainly due to surface deformation or destruction of the metal. In this case the hardness H and its decrease under the effect of adsorption or of surface charging-up with formation of a double ion layer, will be the decisive factor. Attention should be drawn here to the fact that one cannot, by analogy, directly deduce from the reduction of the surface strength of metals due to adsorption that there will be an increase of wear during friction in the presence of surface active media (lubricants) in conditions of high local pressures, i.e. when the tangential stresses set up in the surface layer are considerable[99]. Such an increase of wear is not harmful but is, in practice, found beneficial and is used to speed up running-in (surface finish of machine parts and mechanisms) and for the rapid elimination of local damage on rubbing surfaces producing high local pressures. When the damage is worn away and the surface is smooth once again, the actual contact area increases rapidly; this is associated with a decrease of the normal and tangential stresses in the surface layers. Under such conditions the effect of surface active media on rubbing surfaces consists in a normal friction- and wear-reducing lubricating action.

As pointed out repeatedly by L. A. Shreiner, other things being equal, the value of the adsorption effect is determined solely by the character and intensity of the stresses acting in the surface layer of the solid[24].

Failure to take this important fact into account has often led to misunderstandings[100]. Some investigators attempted to detect an adsorption-induced reduction of hardness by the normal methods impressing a ball-, cone- or pyramid-indentor into the metal surface. In these conditions considerable compressive stresses are set up in the surface preventing the development of weak spots (microcracks) and, consequently, of adsorption effects. The effect of the medium on deformation processes decreases and ceases completely in conditions of hydrostatic compression. The adsorption effect is greatest in the case of fairly large tensile stresses set up in the surface layers.

Adsorption, by assisting deformation, accelerates metallic flow in the surface layers assuring their smoothness, i.e. a high quality surface finish; in other words development of a Beilby layer. This is of considerable importance when working metals by pressure and by cutting, underlining the necessity of using surface active lubricants. On the other hand, as recently shown by G. I. Epifanov[101], the effect of the active medium during metal cutting is not attributable to adsorption assisted metallic flow in the surface layers of the work piece and chip, but rather is expressed in a marked lowering of the specific cutting energy for the metal in the given fluid. This reduction of cutting energy, by a factor 2-5, is, over a fairly wide range of energies, independent of the depth of cutting and not subject to the simple relations between adsorption and the concentration of surface active material: the maximum effect is not found for small concentrations, corresponding to formation of monomolecular adsorption layers on the newly formed metal surfaces, but to the surface active material in the liquid state. The most probable interpretation is that the molecules of the adsorbing material penetrate by two-directional migration along the surfaces developing in the cutting zone; they then, due to the catalytic action of the highly activated clean metal surface, break up, and the resulting atoms or groups of atoms penetrate into the metal lattice. This penetration is associated with considerable hardening and increased brittleness, in other words, with removal of a considerable part of the plastic deformation preceding fracture, and with a considerable reduction of the necessary cutting power. Chemical interaction, between surface active materials and newly formed metal surfaces free from oxide films, can be considerably assisted by limited deformation of the metal, as proved by the experiments of G. I. Epifanov and N. A. Pletenova. This agrees with the published data about the development of metal-organic compounds under similar conditions.

This brief survey shows that extension of research in the field of surface active media to include deformation processes in metals, has led to a number of important conclusions, not only in physical chemistry and the physics of solids, but also in numerous practical fields.

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