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COLLOIDS AND SURFACES A

Physical–chemical mechanics in the studies of Peter A. Rehbinder and his school

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Abstract

In connection with the birthday centennial of academician P.A. Rehbinder, a short review is presented of studies in some principal directions of the physical–chemical mechanics initiated by him and developed by his successors namely: the liquid metal embrittlement observation with the method of electrochemical microscratching; discovery of the reciprocal interaction of solid phase and medium in heterogeneous catalysis and catalytically enhanced sintering; direct experimental study of crystal bridging and residual stresses arising in processes of hydration hardening; and explaining the role of the lyophilic structure–mechanical barrier formed by adsorption layer as a factor of strong stabilization in colloid dispersions. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Disperse systems; Materials; Physical–chemical mechanics; Rehbinder

The year 1998 marks the birthday centennial of the famous scholar and remarkable man, Peter A. Rehbinder (3 October 1898–12 July 1972), my teacher and great friend. This year also marks 70 years since the birth of a new branch in the colloid and surface science created by him: the physical–chemical mechanics of disperse system and materials (in 1928, Rehbinder published the first work on the influence of surface-active medium on mechanical properties of crystals [1]).

Some main notions in this field, the ideas put forth by Rehbinder in its basis, could be illustrated by a scheme[1], Fig. 1. We have to stress the principal common factors that unite disperse systems and materials in this scheme; these are:

(1) Universal importance of their mechanical properties, i.e. resistance to mechanical effects, and our interest in controlling these properties in two opposite aspects, both increasing such resistance (strength, durability), and also decreasing it (for better formation, treatability).
(2) The real, filled with defects physical structure, microheterogenuity, with the highly developed interfaces, both external and internal ones.
(3) Correspondingly, the important or predominant role of various chemical and physical–chemical phenomena at these interfaces, and particularly, of the surface-active components in the ambient medium.
(4) The obvious fact might be underlined that: all *mechanical* processes of deformation and frac-

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[1] This scheme is used in the lecture course on Physical Chemical Mechanics, initiated by Rehbinder in the early 1960's, that the author has been reading since that time annually at Moscow University, and in the last years also at Johns Hopkins University.

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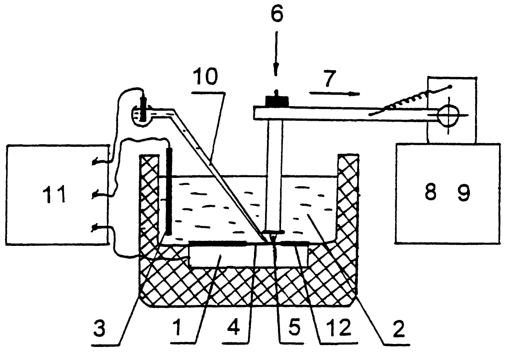


Fig. 1. The scheme of the experimental device for the ECMS tests: (1) sample (cathode), (2) electrolyte solution, (3) anode, (4) open area used for scratching, (5) indenter, (6) direction of the load, (7) direction of the indenter movement, (8) synchronous electromotor, (9) reductor, (10) Luggin's microelectrode, (11) potentiostate, and (12) varnish.

ture are the processes of reconstruction and rupture of interatomic bonds, i.e. essentially *physical–chemical* ones.

(5) Alongside this, disperse systems and materials are united with the common cycle of their transformations, both in technology and in nature. The left and right boxes in Fig. 1 manifest this universal reciprocal connection, and reflect the Rehbinder's principle: "To strength — through dispersion".

In a short presentation, it would be impossible to list all the scientific contributions of Rehbinder in the 50 years of his research activity, and by his disciples and successors over the next 25 years. The information concerning both the development of physical–chemical mechanics, and of other principal branches of colloid and surface science is available in two volumes of the P.A. Rehbinder selected works [2,3], and in a number of proceedings and reviews, e.g. [4–7].

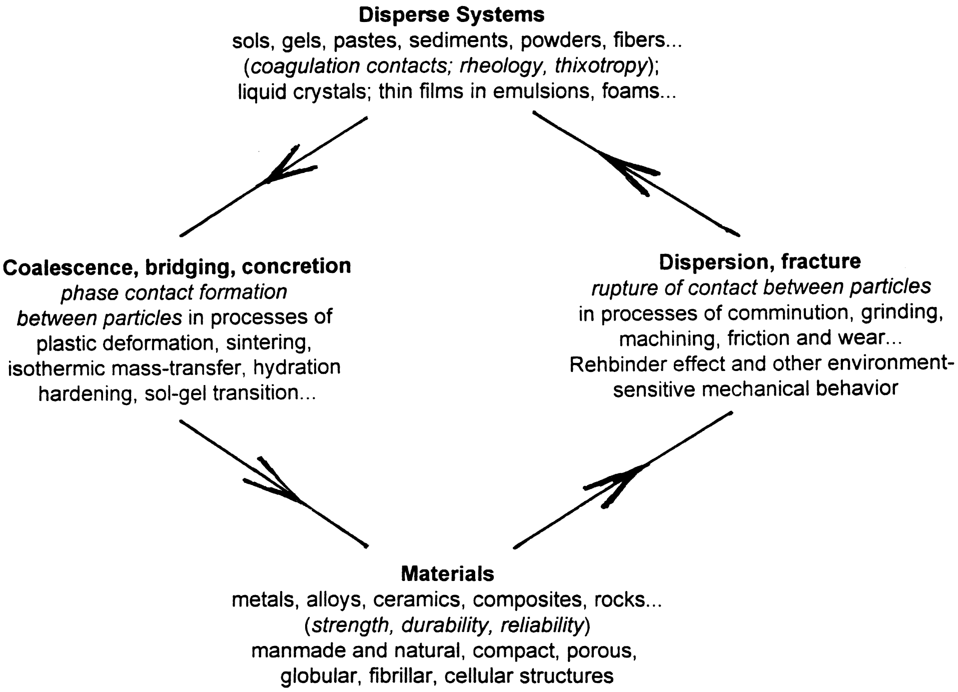
In this report, the contents are confined to only four illustrative pages taken from the research carried out, mainly in later years, in the Institute for Physical Chemistry of the Russian Acad. Sc., and Moscow State University, on the basis of the studies initiated there by Rehbinder.

The author would like to consider this material as the direct development of those studies in some principal directions in the physical–chemical mechanics united by the common approach mentioned above, as examples of the realization of Rehbinder's ideas and foresight.

1. The Rehbinder effect: development of study in the liquid metal embrittlement (LME)

Physical–chemical mechanics was born in 1928 as the first study in the influence of the surface-active medium on the mechanical properties of calcite and some other minerals [1,3]. This phenomenon, the Rehbinder effect, explained as a result of the decrease in the surface (interfacial) energy, was then observed as universal and studied in detail for all types of solids and materials [8]. Effects of the environment-sensitive mechanical behavior of solids (ESMB) are very diverse in their mechanisms and manifestations [8–10]; the most dramatic form is the sharp decrease in strength, and embrittlement of solid metals and alloys contacting some liquid metals [8,11–13]. Such LME effects have been found practically for all metals, including the most strong, and refractory ones . Elucidation of their mechanisms and trends allows one to solve important practical problems: both prevention of damage caused by contact of the stressed material with metal melt (soldering, welding, liquid metal heat carriers in nuclear reactors, etc.), and use of this phenomenon for enhancing machining — drilling, grinding (i.e. dispersion) of the extremely hard metal materials [8].

However, the very presence of the liquid metal phase hinders both studies and applications of the LME effect because of the necessity of elevated temperatures, difficulties in obtaining perfect wetting and reliable contact with the melt, and particularly in removing its residual films or traces, etc. The principal new step in this field is the elaboration of a special method using the LME "*in the absence of the liquid metal phase*", namely, in an electrochemical cell, in the course of cathode reduction of the ions of the selected surface-active metal at the surface of the tested sample, in the minimum and controlled amount, down to monolayers, and at room temperature [14,15]. Two obligatory conditions of the Rehbinder effect can be fulfilled here: the surface activity of the correspondingly



Scheme 1. Physical-chemical mechanics of disperse systems and materials.

chosen component in the electrolyte causing significant lowering of the surface energy of the treated metal (thermodynamical factor), and mobility of active atoms allowing their penetration into the fracture zone (kinetical factor).

In Fig. 2, the scheme is shown of the device for studying stability and damageability of the solid metal surface by the microscratching (microsclerometry) method in such electrochemical cell ("electrochemical microscratching" — ECMS) [14]. In Fig. 3, the results are presented of the ECMS study of the aluminum (99.999%) surface in the solution of gallium salt [15]: the width of the groove in the absence of electrical current, and under cathode polarization of the sample, with the current density of 6.8 mA cm^{-2}. In such a case of the very high plasticity of the tested metal and relatively soft stressed state in the contact zone, the decrease in mechanical resistance is manifested not by embrittlement, but by the *plasticizing* effect [8]. Under this effect, the broadening of the groove reaches 20% and increases with lowering the load applied to the indenter, i.e. with the localization of deformation in the thinnest subsurface layer. Similar observations have been carried out on gold, with cathode deposition of mercury. However, in the

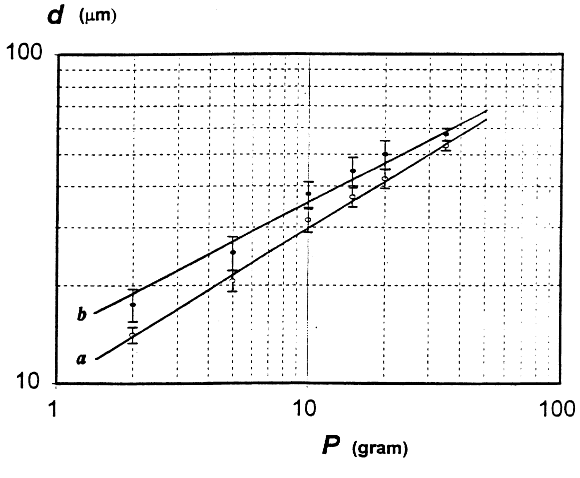


Fig. 2. The dependence of the microscratch width d (µm) upon the indenter load P (gram): in the absence of the current (a), and under cathode polarization of the surface, with the current density of 6.8 mA cm^{-2} (b).

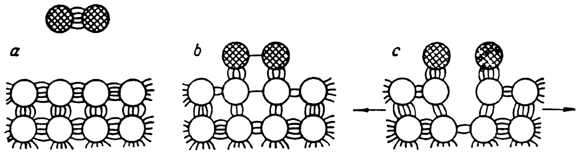


Fig. 3. Scheme showing simultaneous decrease in the strength of interatomic bonds in a solid and in medium molecules as a result of their reciprocal influence during adsorption (a) and (b), and caused by this influence facilitation of the solid fracture under the effect of applied stress (c).

case of the high intrinsic hardness of the material, e.g. titanium aluminide, an active component (say, cadmium) causes brittle cracks to occur. The possibility to differentiate these two manifestations in the environment-sensitive mechanical behavior (ESMB) is important for optimizing processes of friction, wear end lubrication.

This method allows us to study also the influence of the reduction of non-metal cations, e.g. the known effect of ammonia on mechanical behavior of brass, alongside with the anode oxidation effects, like stress-corrosion, etc.

The cathode reduction of active metals (cadmium, zinc) has been used, instead of their liquid phase, for enhancing the effectiveness of grinding the highly tempered steel [16].

2. The reciprocal influence of solid phase and medium in heterogeneous catalyses

In the early 60s, P.A. Rehbinder and the author were involved in the problems of the strength and durability of catalysts [17]. It occurred that one of the main causes for fracture of granules was also the Rehbinder effect: the combination of the active components adsorption (chemisorption) and mechanical stresses (both external, and internal residual stresses), as was shown in the direct experiments with the MgO, Co–Mo, Al–Cr–K, and other catalysts [18,19]. In collaboration with industry, ways and methods were found to optimize manufacturing of a number of leading catalysts (in oil cracking, caoutchouc monomers, ammonia synthesis), by means of perfecting contacts between particles and reducing residual stresses.

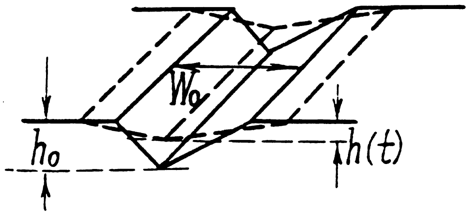


Fig. 4. Scheme of evaluation of the surface diffusion coefficient D_s by measuring decrease in the depth $h(t)$ of a microscratch with the initial width W_0: $D_s = \text{Const.}\, W_0^4\, d/dt\, [(h/h_0)^{-4/3}]$.

An important fundamental result of these works was the formulation of the principle of the reciprocal influence of the solid surface and medium in the heterogeneous catalytic processes, as illustrated by Fig. 4 [18]. For example, iron is well known as a catalyst in ammonia synthesis, taking part in the dissociative adsorption of hydrogen, and the reciprocal effect is the hydrogen embrittlement of steel. Similar phenomena take place for nickel as catalyst of the benzene hydrogenation, etc. The calculations show that the energy of an elementary event of such (exothermic) reactions can be enough for rupturing bonds in the lattice of solid phase, and for escaping of an atom from the lattice to the surface, i.e. transformation to the state of *adatom* participating in the surface self-diffusion. Examples of the reconstruction of the catalyst surface were known earlier and were considered as catalytic corrosion [20]. The direct measurements using the method of the microscratch healing (Fig. 5) showed the increase in the surface self-diffusion

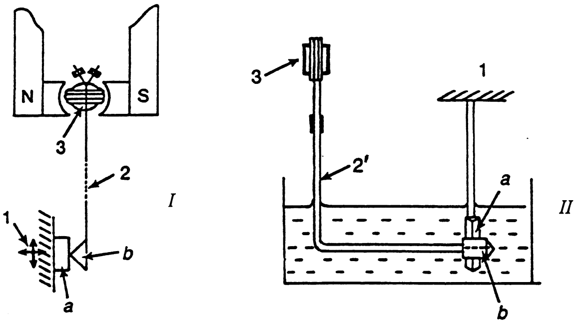


Fig. 5. Device for the measurements of the cohesion forces in contacts between particles (I — in air, II — in liquid): (1) manipulator, (2) holder, (2′) special L-shaped holder, (3) magnetoelectric dynamometer; *a* and *b* — samples (crystals).

coefficient up to 10–100 thousand times, in both the mentioned cases with iron and nickel [18,19], and namely at the expense of the increase in the adatoms concentration (temperature of the metal surface did not change).

This effect opens way for the essential facilitation of processes of metal powder sintering. In Table 1, data are presented concerning the increase in the strength of the pressed powder samples of the iron catalyst in the course of CO oxidation, in comparison with other media, at the same temperatures [21]. This significant, up to six-fold or more, effect is immediately connected with the growth of contacts between particles due to the accelerated surface mass-transfer. In turn, this gives opportunities for the significant lowering of the sintering temperature providing the same strength of contacts and material as a whole: in the case considered, it was about 300°C. Similar data were found, with the same catalytic reaction, for nickel powder samples.

Recently, these studies were also extended to oxide systems, and corresponding results in the catalytically enhanced sintering (CES) have been obtained for alumina and zirconia [22].

3. Crystallization structure forming

Many years of the research activity of Rehbinder and his disciples and co-workers in Moscow University and other institutions were devoted to mechanisms and trends in formation of space structures during transition of the disperse system, with its mechanically reversible and weak (coagulational) contacts between particles to material, with strong "phase" contacts [3,23]. The works on catalyst strengthening mentioned in previous page also belong to this cycle.

Special attention in this field was paid to the hydration hardening of mineral binders, the primary physical–chemical process in the technology of the main building materials — cement and concrete [24–27]. In Rehbinder's works, widely recognized as an essential contribution to this area, it has been shown that the predominant mechanism of hardening consists of nucleation of a new hydrate phase in the solution of an initial phase supersaturated with respect to new formations, and bridging of these new phase particles. Kinetics of the changes in rheological parameters, of the strength growth (and drops), was studied in details, in dependence upon the complex of conditions, particularly, supersaturation curve, and hypothesis was spoken on the possible role of internal stresses in structures obtained.

However, one of the principal factors in such a process — conditions of crystal bridging — was not clear. The author took part in the discussion between P.A. Rehbinder and N.N. Semenov when the latter formulated the question especially vividly: "Why do they bridge, despite the repulsive forces (electrical double layer, crystallization pressure)?" This principal physical–chemical problem initiated direct experimental study, with the first results presented for publication in the Doklady AN SSSR by Semenov, after Rehbinder's death [28].

The method is shown in Fig. 6: two crystals, e.g. gypsum (calcium sulphate dihydrate), are brought into contact in the aqueous solution of calcium sulphate supersaturated with respect to the crystals solubility in the ratio $\alpha = 1–4$, for the time $t = 10$, 100, 1000 s, under compressing force $f = 0.1$, 1, 10 dyne. Histograms of the measured then rupture

Table 1
Calculated values of the enthalpy of mixing H_m (eV) and interfacial energy σ_{sl} (mJ m^{-2}) for some solid–liquid metal couples, and data on the LME effect: experimentally observed (+) or absent (−) in these systems

Solid–liquid	H_m	σ_{sl}	LME
Al–Zn	0.16	60	+
Co–Bi	0.01	110	+
Cu–Bi	0.17	220	+
Mo–Au	0.74	580	+
Mo–Hg	3.14	1980	−
Nb–Cd	2.57	1470	−
Nb–Sn	0.22	240	+
Ti–Cd	0.28	260	+
Ti–Ga	−1.79	(−860)	−
Ti–Hg	0.06	140	+
Ti–Pb	−2.73	(−1380)	−
W–Hg	3.75	2380	−
W–Pb	0.39	420	−
Zn–Ga	0.04	110	+
Zn–Sn	0.08	150	+
Zr–Bi	0.94	500	+

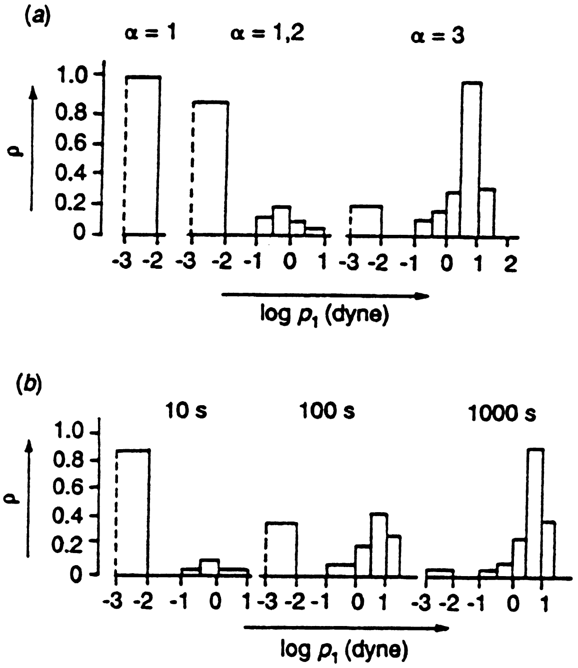


Fig. 6. Differential distributions of contact strength p_1 emerging between gypsum dihydrate crystals brought together under force $f=10$ dyne in calcium sulphate solutions: (a) for various supersaturations $\alpha(t=100$ s); (b) for various time of contact t $(\alpha=1.8)$; ρ is distribution density.

force, i.e. the contact strength p (Fig. 7) reveal the obvious differentiation between the initial weak (coagulational) contacts and the newly formed strong (phase) ones. The dependence of the probability (share) w of the latter upon α and t (Table 2), elucidates the fluctuational mechanism of the primary nano-size nuclei bridge in the contact zone arising, followed by its quick spreading over some microscopic area. This model allows one to evaluate the activation energy of such nuclei contact formation (of the order of 10 kT), and its size (of 1 nm).

The newly found factor in the crystal bridging occurred the obligatory presence of the *compressive force*, as this is seen in the dependence $w=w(f)$ in Table 3. In the further development of these works, the peculiarities of the crystal bridging were studied for various crystallographic orientations, for crystals of a given mineral with different ones, under varying pH, ionic strength, surfactant additives, etc. [29]. In all cases, the condition of mechanical compression remained obligatory.

Along with this, the flip-side of such a factor is revealed: its memory in the obtained material structure as residual internal microstresses, i.e. metastability, the a priori factor of the following decrease in the strength and durability of the material, particularly, under the influence of active media.

The special precise X-ray method allowed, for the first time, observation and measurement of the value of such residual microstresses (so-called, Modus II stresses), and also of macrostresses arising in the course of the dehydration of samples (Modus I stresses). It occurred that they reached tens and hundreds of MPa, i.e. the level of the material strength. Experiments with additives of various surface-active substances, both hydrophilic and hydrophobic, revealed their essential influence on the value of residual stresses at different stages of hydration, thus opening opportunities for the process optimizing [29].

4. The structure–mechanical barrier

In their large cycle of the works, Rehbinder and colleagues developed ideas concerning the *strong factors of stabilization* of disperse systems (sols, emulsions, foams) as providing stability against coagulation and coalescence, under conditions of high concentration of the disperse phase, high salinity, temperature changes, etc. [30]. The typical examples are the interfacial adsorption layers (elementary membranes) in dairy products, crude petroleum, natural latex, and also the walls of living cells. In these cases, the classical thermodynamical factors (DLVO electrostatic barrier, thin films elasticity after Gibbs), kinetical (Marangony–Gibbs effect), or hydrodynamical ones (Reynolds flow) are not enough. After Rehbinder, the two obligatory conditions of such strong stabilization are: the *mechanical strength* of the adsorption layer (in some sense, a “non-thermodynamical” factor), and the *lyophilicity* of this adsorption layer with respect to ambient dispersion medium. These factors take place in the

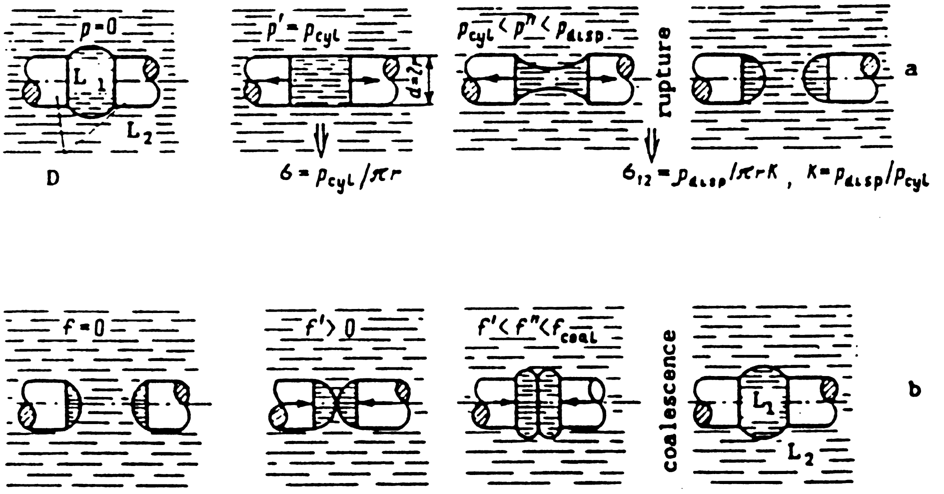


Fig. 7. Graph (a): rupture of liquid drop L_1 placed between cylindrical holders D with diameter $2r$ in dispersion medium L_2, when subjected to a tensile force p. The force p_{cyl} at which the drop assumes the form of a cylindrical column gives the value of the interfacial tension $\sigma_{12}=p_{\mathrm{cyl}}/\pi r$; with increasing p_{cyl}, the rupture force p_{disp} also increases. Graph (b): coalescence of two previously formed drops under the influence of a compressive force f. The force f_{coal} characterizes the mechanical resistance to coalescence.

effects of typical protective colloids, particularly, biopolymers.

With the immediate participation of Rehbinder, and in further works in the Colloid Chemistry Department of the Moscow University, the direct experimental studies of these both factors were carried out: rheological characteristics, including strength of interfacial adsorption layers of various low- and high-molecular surfactant were studied [31], and measurements of the free energy of interaction, as the principal parameter of the surface lyophilicity, have been performed for diverse hydrophilic and hydrophobic surfaces in polar and non-polar liquids, and in surfactant and

Table 2

The strength P (MPa) of iron catalyst after catalysis (CO oxidation), and after annealing in various media. The rate of throughput of reactants of 100 ml min^{-1}; the initial strength of samples of 1.3 MPa; porosity of samples is 57.5%

Composition of reactants	P (M Pa)	
	573 K	623 K
8% CO+4% O_2+88% He (catalysis)	6.5	7.4
Air	2.4	2.9
Argon	1.4	1.5
8% CO+92% He	0.3	0.4
H_2	—	0.9

Table 3

Probability of bridging W_b of $CaSO_4 \cdot 2H_2O$ crystals in supersaturated calcium sulphate solutions as a function of compressive force f, supersaturation α, and time of contact t

	W_b (%)											
	$\alpha=1.2$			$\alpha=1.5$			$\alpha=1.8$			$\alpha=3.0$		
	f (dyne)			f (dyne)			f (dyne)			f (dyne)		
t (s)	0.1	1	10	0.1	1	10	0	1	10	0	1	10
10	0	0	0	0	1	5	0	2	7	0	7	38
100	0	4	17	0	8	24	1	14	49	4	53	83
1000	4	22	33	7	44	68	10	60	96	33	91	100

Table 4
Stability of drops of nonpolar liquids (heptane, perfluordecaline, perfluormethylcyclohexylpiperidine, perfluorthreebuthylamine) in aqueos solutions of Pluronic F-68, 5.10^{-9} M (A), and of φ = PEG (perfluordiisononenylpolyethyleneglycol), 5.10^{-6} wt% (B), and values of the free energy of interaction of hydrophobic surfaces (methylated glass) in the same liquids (C)

	Heptane	PFD	PFMCP	PFTBA
A				
^{f} coalesc (10^{-5} N)	<0.01	2.5	9	>10
$\Delta\sigma$ (mJ m^{-2})	15	17	12	10
B				
^{f} coalesc, 10^{-5} N	1.2	0.1		
$\Delta\sigma$ (mJ m^{-2})	4	14		
C				
Nonpolar liquids	$F_{si}/2$ (mJ m^{-2})			
PFTBA	7.0			
PFMCP	6.8			
C_7F_{16}	5.2			
PFD	4.8			
n-C_7H_{16}	<0.01			
n-C_8H_{18}	<0.01			

polyclectrolyte solutions [5,31–33]. In juxtaposition with the data on the stability of corresponding emulsions, these results confirm fully the accuracy of notions about the decisive role of the Rehbinder's structure-rheological lyophilic barrier introduced much earlier than the "steric factor" appeared in the western literature. The latter, in its initial meaning, presents only the part of the elastic resistance of the interfacial adsorption layer, namely "thermodynamical" provided by the conformational entropy of the adsorbed macromolecule loops and tails, similar to "tentacles" in the surrounding liquid phase. Of course, this factor takes place. However, the following data show that its role can be not significant.

A series of our works was devoted to the stabilization of perfluorcarbon emulsions, in connection with the artificial blood substitutes [33,34]. Among other approaches, the direct observations of the resistance of the interfacial adsorption layer to fracturing and expelling were carried out using our method of the rupture of individual droplets, and closing and coalescence of two half-droplets. Compressive force *f* under which coalescence takes place is a very sensitive and illustrative characteristic of the strength of the stabilizing adsorption layer.

In Table 4, the data are presented on this "force of coalescence" for droplets of various perfluorcarbon liquids, and, for comparison, also for hydrocarbon droplets, in aqueous solutions of pluronic, i.e. "regular" hydrocarbon surfactant, and of the fluorinated (φ-PEG (perfluordiisononenylpolyethyleneglycol). Under given conditions (concentrations), the resistance to coalescence of hydrocarbon droplets stabilized by fluorinated surfactant (a), and of fluorocarbon droplets stabilized by "regular" surfactant (b) occurs in one or two orders of magnitude higher than that of fluorocarbon stabilized by fluorinated surfactant (c), and hydrocarbon stabilized by pluronic (d). In comparison with the data on the changes in the interfacial tension, similar in all these cases, and on the free energy of interaction in corresponding systems, significantly different ones, this means that the major resistance to coalescence is not due to the free tentacles [items (c) and (d)], under the full affinity of nonpolar phase and nonpolar parts of surfactant molecules, but due to their *expelling* from the less akin nonpolar liquid phase, and formation of the relatively dense and, correspondingly, mechanically stronger layer at the interface.

The described investigations of the structure–mechanical properties, specifically, strength of the interfacial adsorption layers, and, in general, studies in the broad field of the physical–chemical mechanics of disperse systems and materials find fruitful practical applications in many areas of industry, agriculture, medicine, as was undisputedly shown and promoted by P.A. Rehbinder [2,3].

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