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Experimental Study of Chemical Processes under Dynamic Compression Conditions

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The results are given of studies on the creation of a set of systems for the preservation of specimens of condensed substances subjected to dynamic pressures with different types of stress applied to the specimens: with single and stepwise shock compression and also with nearly isentropic compression. The conditions in the compression of substances in protective tubes using cylindrical and planar compression schemes are examined. Examples of various chemical processes under these conditions are given, attention being concentrated on the reactions of organic substances.
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I. INTRODUCTION

New methods for the generation of high pressures in a condensed medium, based on the use of powerful shock waves (SW), have been proposed and developed in recent years. In particular, shock waves have been generated in the test substance by the action of detonation waves from a chemical explosive on a specimen in contact with the explosive charge or as a result of impact by bodies accelerated to high velocities by the detonation products of the explosive. In condensed (solid or liquid) explosives, up to 8×10^6 kJ m^{-3} of chemical energy can be concentrated per unit volume; the energy is liberated under detonation conditions, i.e. in the course of the ultrasonic propagation of the front of an exothermic chemical reaction in the explosive. After the sharp increase of pressure at the shock wave front, the pressure diminishes owing to the chemical process in the reaction zone. At the end of the reaction zone the detonation products expand and are accelerated in the direction of propagation of the reaction zone. The mixture of gaseous products at the end of the reaction zone has a density of up to 2.5 g cm^{-3}. The detonation wave propagates at a constant velocity, so that the energy losses by the SW are continuously made good by the energy liberated as a result of the chemical reaction in new portions of the explosive.

The detonation wave excited SW in the adjoining medium whose intensity depends on the power of the explosive and the dynamic rigidity of the medium $\rho_0 D$, where ρ_0 is the initial density of the material of the specimen and D the velocity of the SW front. For example, in the detonation of an explosive at a rate of $\sim8\times10^3$ m s^{-1}, pressures of ~45 GPa develop in a steel bar in contact with the charge. Experiment shows that, when a steel plate is accelerated by the detonation of the same explosive, its impact against steel can generate pressures in the specimen which are three times as great.

The behaviour of gases and condensed substances on compression by means of SW shows significant differences. When SW are propagated in a gas, even low pressures induce a marked compression and a considerable increase of temperature by virtue of the high compressibility of the gas. The pressure in the gas has a thermal origin and is always proportional to the temperature. At high temperatures various physicochemical processes occur in gases: excitation of molecular vibrations, dissociation, ionisation, and chemical reactions.

On compression by SW, a condensed substance behaves differently, because the repulsive forces between atoms must be overcome in order to increase density. For this reason, an internal pressure of non-thermal character arises on shock compression in addition to the "thermal" pressure. With increased compression, the first component of the pressure associated with the thermal movement of atoms increases; this leads to limiting degrees of compression. This does not occur under isentropic or isothermal compressions, where an increase of pressure always entails an increase of density.

The first studies showed that the use of shock compression is productive in the study of dynamic compressibility (shock adiabats) of metals, the determination of null isotherms, and the derivation of semi-empirical equations of state in the range of pressures up to several hundreds of GPa and temperatures of ~10^4 K.[1,2] We may note that a break in the shock adiabat for iron has been noticed for the first time[3] at 13 GPa which is the first evidence for a possible change in the structure of the substance during shock compression.

The methods used to measure shock adiabats are based on the measurement of the velocity of the wave front D and the mass velocity of the test substance behind the front u (in practice measurements are made of the velocity of the free surface of the specimen, which is close to twice the mass velocity). Knowing these kinematic parameters of the SW, it is possible to determine the thermodynamic characteristics of the substance (its density $\rho = 1/V$, the pressure P, and the increase in specific internal energy ΔE) from the laws of conservation of mass, momentum, and energy:

$$\rho=\frac{1}{V}=\rho_0\frac{D}{D-u},\quad P=\rho_0 Du,\quad \Delta E=\frac{1}{2}P(V_0-V)$$

D and U are measured with the aid of electrical contacts in the central part of the specimen which is unaffected by stress relaxation waves (for the details of the method, see Al'tshuler's review[1]).

Careful investigations of the dynamic compressibilities of complex inorganic compounds have been carried out at the Institute of Chemical Physics of the USSR Academy of Sciences since 1957. Many such compounds have very high dynamic yield points—up to 10 GPa and above. The propagation of elastic waves with such amplitudes precluded the investigation of the compressibilities of such substances in the monolithic state over a wide pressure range by the methods known at the time, based on the measurement of the velocities of the SW and of the free surface. A "mixed" method,[4] based on the study of the compressibility of a mixture of particles of the test substance in a plastic medium, for example, paraffin, the shock adiabat for which is known, was therefore proposed and applied. This method made it possible to investigate the volume dynamic compressibilities of stable substances such as crystalline quartz,[5] glass,[6] and feldspar[7] also at low pressures.

In order to investigate the complex structure of the SW front in the elastic-plastic region or in phase transitions, an electromagnetic method,[6] previously employed to measure the velocities of the detonation products, was applied. This method made it possible to follow the evolution of the complex wave structures as they are propagated in glass,[6] potassium bromide and chloride,[8] and fused quartz.[9]

The investigations showed that all the substances mentioned above as well as marble[10] undergo structural changes during compression, i.e. in a period shorter than 10^{-6} s. The study of the impact compressibility of graphite[11] demonstrated that this important substance exhibits an anomalous compressibility at pressures of 20–40 GPa, which was interpreted as its conversion into diamond. The break at 60 GPa, caused, according to the authors,[11] by the transition of carbon to a metallic state, is a result of a methodological error, which was demonstrated in later investigations, for example, those of Pavlovskii and co-workers.[12,13] This erroneous result served as a basis for the construction of a phase diagram for carbon[14] in the region of high parameters which unfortunately has been used in the past by many investigators.

The aim of the above method was not to preserve the specimens: after recording a particular parameter, the specimen of the test substance disintegrated and was lost. For this reason, although these methods do in fact yield information about the pressure range in which the state of the test substance changes, about the structure of the SW front, and about the change in the electrical and optical parameters of the test substance, we obtain in essence only indirect data about the reactions which the test substance has undergone. These reactions are responsible for the anomalous behaviour of the shock adiabats observed for a whole series of substances.[3–16] In the first studies[3–11] it was already found that the anomalies on the shock adiabats, associated with the possible structural transformations in the test substances, occurred at relatively low pressures in the range 10–40 GPa, at which the preservation of the specimens was entirely possible; further studies were therefore developed with the aim of creating special devices (preservation tubes).

The difficulty of preserving the specimens after shock compression, particularly at ultrahigh pressures, is due to the presence in the decompression waves of powerful extending stresses which lead to the rupture of metallic containers and the loss of the test specimens. In constructing the retaining devices, the main task is to discover the region of the working volume within which the decompression waves do not interact and there is no appreciable macroplastic deformation. The appropriate positioning of the tubes with the specimens largely determines the success in devising preservation methods.

The first studies on substances preserved after impact compression were published at the end of the 1950's. In the first place one should mention the investigation[17] of the physicochemical transformations of substances under the influence of SW. This communication describes experiments on the shock compression of substances placed in cylindrical preservation tubes. It was shown that compression resulted in the partial decomposition of paraffin, NaCl, $CuSO_4$, $MgCO_3$, and $Pb(NO_3)_2$; on the other hand, sulphur underwent a phase transformation. Other investigations of preserved substances revealed the decomposition $Fe_2O_3 \rightarrow Fe_3O_4$ with decrease in the valence of the metal[18] and the conversion of SiO_2 into an amorphous form.[19] An important result has been obtained:[20] after the shock compression of graphite in a cylindrical tube, a small amount of finely dispersed diamonds was isolated. The results of investigations of metallic specimens (iron, steel, copper, and aluminium) and certain other technically important materials, preserved after shock compression, have been published.[21,22] Studies on the strengthening and welding of metals by an explosion have also begun to develop since 1960 (see the relevant monograph[23]). Much attention has been devoted to the explosive pressing of powdered materials.

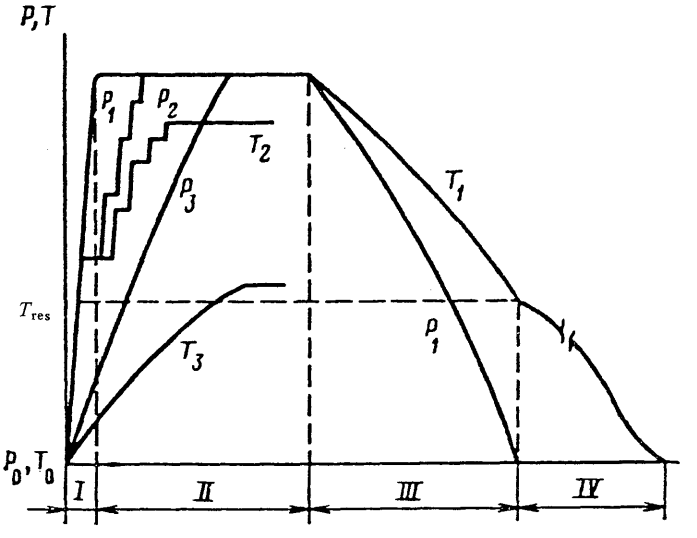


Figure 1. Idealised time variations of P and T at a certain point in the specimen in single (P_1, T_1) and multiple (P_2, T_2) shock compression and in dynamic isentropic compression (P_3, T_3). The rate of cooling of the substance along section III is not less than 10^8 K s^{-1}.

In the studies quoted the experimental details and the conditions in the shock compression of specimens as well as the degree of macroplastic deformations of the tubes were inadequately described. The character of the interaction of the SW with the test substance had not yet been investigated at that time.

For a better understanding of the questions considered in this review, we shall deal with the general character of the changes in the pressure and temperature in the specimen in experiments with preservation, i.e. when the specimen is acted upon by the entire shock compression cycle, including rarefaction waves.

Fig.1 presents the idealised temperature and time variations of the pressure under the conditions of dynamic compression, by which we understand the compression of the test substance subjected to different types of stress; from compression by single-step and multistep SW to dynamic isentropic compression (DIS) or more precisely compression approaching isentropic compression. In the case of a single compression of the test specimen, the P and T increase simultaneously and very rapidly (over a period of 10^{-12} and 10^{-9} s) in the SW front (region I); the compression phase continues for 10^{-6} s (region II). The pressure is then removed by the isentropic decompression waves and (owing to the irreversibility of the shock compression) the temperature diminishes to a certain value T_{res} (region III). Further cooling of what is now an unstressed specimen takes place in accordance with the usual thermal conductivity laws (region IV). The high residual temperatures in region IV can lead to the liquefaction (annealing) of the phase formed on compression and (or) to the occurrence of chemical reactions.

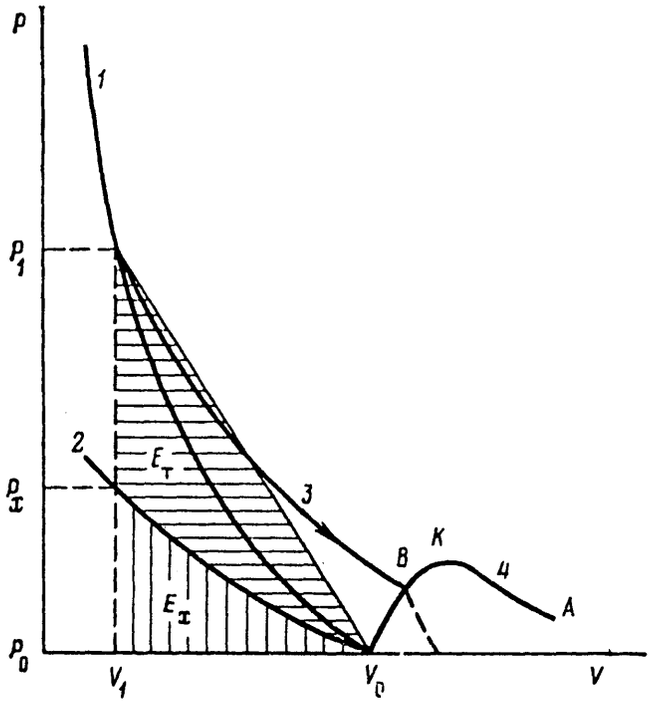


Figure 2. The $P-V$ relation on shock compression of the test substance and its relaxation: *1*) shock adiabat; *2*) cold (elastic) compression curve; *3*) rarefaction adiabat. The shaded regions correspond to the cold (E_c) and hot (E_h) components of the total energy of the compressed specimen; *4*) curve separating the regions of the single- and two-phase states of the substance.

Fig.2 illustrates the shock adiabat (curve *1*), the cold compression curve (curve *2*), the decompression adiabat (curve *3*), and curve *4* separating the regions of single-phase and two-phase states of the substance; its V_0K branch before the critical point K represents the boiling point curve (the onset of vaporisation) and the branch KA represents the saturated vapour curve (the onset of condensation).

If the amplitude of the SW is not high, then, as a result of compression, the test substance passes from the initial P_0V_0 state to, for example, the state P_1V_1 (Fig.2). After decompression, the states of the system follow the isentropic decompression curve [shown by an arrow in Fig.2 (curve *3*)], which intersects the boiling point curve at point B, after which the solid should, in principle, boil. However, the rate of this process is negligible at temperatures of the order of hundreds and even thousands of degrees (for metals) and in reality the solid expands and cools to zero pressure along the "superheated liquid" adiabat shown dashed in Fig.2.

If the SW is very powerful and the internal energy of the heated substance is many times greater than the bond energy between the atoms (equal to the heat of vaporisation at $T = 0$), then, following its expansion to $P = 0$ in the region below the point K, the substance can completely vaporise. This problem has been analysed in detail in a book[2] (p.550).

Various designs of the preservation tubes for the dynamic compression of condensed substances with the aim of carrying out various physicochemical transformations are examined below. Attention has been concentrated on the conditions in the compression of the test substance following the application of different kinds of stress; certain results illustrating the possibility of the method based on dynamic influences are presented.

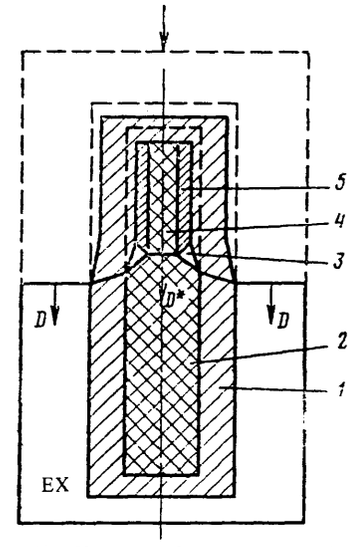


Figure 3. Schematic diagram of the compression of a substance in a cylindrical tube; D = rate of detonation of explosive (EX); D^* = rate of propagation of head SW: *1*) metallic tube; *2*) test substance; *3*) and *4*) regions of single compression behind the front of the incident and head SW; *5*) region of double compression behind the front of the incident and reflected SW. The dashed lines indicate the initial positions of the explosive, the tube, and the specimen.

II. THE BEHAVIOUR OF SUBSTANCES COMPRESSED BY CONVERGING WAVES

Cylindrical preservation tubes are the principal means of carrying out dynamic experiments for chemical purposes. Fig.3 illustrates a cylindrical metallic tube and the test

substance placed in it; the tube is arranged along the axis of the explosive charge. As a result of the detonation of the explosive, the detonation wave passing around the cylinder excites a conical converging shock wave in it and in the test substance. The wave undergoes a transition to a three-shock configuration which is propagated in the stationary region at a rate equal to the rate of detonation of the explosive employed.[24] The three-shock configuration arises in solid (continuous and porous) substances and in liquids.

The details of the investigation have shown[25] that there is a linear relation between the diameter of the head wave (the Mach wave), occupying the central part of the three-shock configuration, and the diameter of the specimen. It is significant that in the central part of the tube the test substance undergoes a single shock compression by the head wave, while at the walls of the tube it undergoes a double compression by the incident and reflected SW; as a result of this, for the same final pressure, the shock heating of the test substance in the centre of the specimen is much greater than at the walls of the tube. The compression conditions also differ along the length of the specimen. It is almost always possible to differentiate three zones: the region where a three-shock configuration is produced, the region of the stationary propagation of the wave, and the region at the bottom of the tube where additional compression of the test substance by the SW reflected from the bottom can take place. Thus in a single experiment the substance located in different zones of the cylindrical specimen is subjected to stresses of different kinds.

The observed relation between the rate of propagation of the three-shock configuration and the rate of detonation of the explosive charge employed made it possible to determine the pressures developed without special direct measurements (when the shock adiabat for the test substance is known). It was found that it is very simple to achieve pressures of several hundreds of GPa. Thus the use of an explosive whose detonation velocity is $\sim 8 \times 10^3$ m s^{-1} generates maximum pressures of ~500, 60, and ~35 GPa in monolithic specimens of tungsten, sodium chloride, and perspex respectively. In order to increase the pressure, for example, in salt, it is sufficient to mix and compact tungsten and salt powders. With a low sodium chloride content, pressures close to 500 GPa can be reached as a result of repeated compression and equalisation of the pressure in the specimen.

The next important factor was the occurrence of very intense plastic deformations in the working volume of the cylindrical tubes with axial filling.[26] The particles of the test substance can migrate under such conditions relative to one another over considerable distances in the direction of propagation of the three-shock configuration, particularly in the central part of the tube. Evidently such conditions should promote the decomposition of chemical compounds, should significantly accelerate the fusion processes, and should facilitate chemical interactions of substances. An attempt has been made to separate the decomposition products using intense plastic flows.[26] For this purpose, a very small amount of the test substance (0.5 wt.%), for example, an oxide, was mixed with ground quartz and the mixture was subjected to shock compression. After the experiment the metallic shell was dissolved in hot hydrochloric acid and the remaining monolithic quartz bar was ground and polished. The ground sections were investigated under a microscope. This method revealed traces of the movement of the particles of the material of the tube into the test specimen and also traces of plastic deformations shown by a change in the colour of the test substance following the chemical reaction. In order to protect the test substances from contamination, quartz or glass inserts were used, which completely prevented the migration of the contaminant into the specimen. It was shown in these experiments that, during the plastic flows in the preservation tubes under shock compression, the particles of the test substance can migrate in the direction of the SW over a distance of 2–3 and more centimetres.

The behaviour of more than 60 oxides has been investigated by this method under identical shock compression conditions. Special measurements showed that in porous quartz ($\rho_0 = 1.48$ g cm^{-3}) and hence also in the test oxides a pressure of ~48 GPa developed; the concentration of energy was then $E = 1.2 \times 10^4$ kJ kg^{-1}. The capacity of oxides for decomposition under shock compression was determined by the enthalpy ΔH^0_{298} of the process involving the elimination of one gramme-atom of oxygen. Oxides with ΔH^0_{298} ranging from 62.8 kcal g-atoms^{-1} (Ag_2O) to 211.0 kcal g-atom^{-1} (CaO) were investigated. It was found that the decomposition limit of the substance subjected to the influences indicated corresponds to a quantity which is only 20% below the overall energy of the shock compression.[26]

In order to ensure the chemical purity of the experiments, it was necessary to determine the degree of contamination of the test substances by the materials of the walls of the tubes as a function of the properties of the substances and the conditions of the dynamic influence. For this purpose, the contamination of sodium chloride and alumina, placed in steel-65 tubes, was determined by chemical analysis. Sodium chloride was used in the form of a powder or was fused *in vacuo* on the inner part of the tube. After the experiment, the tube was opened, the test substance was extracted, and the distribution of iron along the length and radius of the tube was investigated. The results showed that, when the test specimen is monolithic, then the material of the walls hardly penetrates the test substance. On the other hand, if the tube is filled with a powder, particularly one which is difficult to compact, such as Al_2O_3 (in the experiments the initial density of Al_2O_3 was 1.1 g cm^{-3}, while that of powdered NaCl was 1.9 g cm^{-3}), then the degree of contamination of the specimen by iron reached several tenths per cent.[27] In the above study it was shown that, if for any reason, it is not possible to employ a monolithic specimen, then one can subject the specimen to preliminary impact compression using the detonation of a low power explosive charge which causes only an insignificant contamination of the test substance but ensures its effective compaction. At the same time a sharp decrease of the maximum temperature developed on repeated compression is achieved, because the specimen had been subjected to preliminary compaction.

The influence of SW on porous substances as a rule leads to the formation of more or less monolithic cylinders having in most cases a characteristic structure: along the axis, there is a filamental cavity 0.1–0.5 mm in diameter. Its formation is due to the interaction of the rarefaction waves. A cavity 1–3 mm in diameter is sometimes formed in the lower part of the specimen.

Under certain conditions (high pressures and a low crystal lattice energy of the test substance), the substance may not only melt but may evaporate at the centre of the tube. The migration of the test substance in the direction opposite to that of the propagation of the SW has been demonstrated by the radioactive isotope method.[28]

After the completion of the process, a characteristic microstructure, usually arising during the radial crystallisation of the fused metal in the tube during its uniform external cooling, can arise throughout the volume of the cylindrical specimen. This feature of the shock compression of the test substance in cylindrical tubes is associated with the difference

between the residual temperatures of the substance and the walls of the metallic tube. This difference is particularly large in the study of specimens in a porous state where the residual temperature of the substance can reach several thousands of degrees, while that of the metallic tube is hundreds of degrees. As a result, conditions are created for the rapid cooling and quenching of the test substance. For example, estimates for niobium pentoxide have shown[29] that the total crystallisation time of the melt is ~0.3 s, while the linear rate of crystallisation is 0.05 m s^{-1}; for tungsten, these quantities are 0.005 s and 0.3 m s^{-1} respectively.

In most cases specimens with a reduced density, whose pores contain air at atmospheric pressure, are investigated in cylindrical tubes. One can assume that during the shock compression of such systems with a small particle size k, the pressure is equalised but thermal equilibrium is not established. Owing to the small weight fraction of air, its contribution to the temperature is small and the average temperature of the mixture differs little from that of the solid component, although in the initial instant of the compression the air in the pores was heated to a much greater extent. After intense shock compression, the porous medium undergoes a major volume deformation and hence is heated more than a continuous substance compressed to the same final pressure.[1] Even after the shock compression of porous specimens subjected to preliminary evacuation the temperature developed may amount to tens of thousands of degrees and part of the substance passes to the state of plasma;[30] thermal emission of electrons can take place under these conditions.[31]

The shock compression of porous substances does not entail major difficulties in the solution of the problem of the preservation of the compression products. Difficulties arise when the test substances decompose or react with the evolution of a large amount of gas and especially when the pores have been filled beforehand with gases which have been compressed to high pressures or liquefied. As a rule, the usual tubes are ruptured under these conditions by the high internal pressure. In order to preserve the tube without rupture, it is necessary to develop special designs. Evidently the preservation of the tube should be promoted by the situation where the gas reacts partly or fully with one of the components of the specimen on compression to form a solid product. It has been shown[27] that the compression of the oxides, fluorides, chlorides, iodides, and sulphates of univalent, bivalent, quadrivalent, and hexavalent elements did not lead to the rupture of the tubes. Comparatively readily decomposed substances such as copper sulphate, barium chloride dihydrate, paraffin, phosphorus oxide nitride, and nickel fluoride hydrate and substances which are highly volatile under the usual conditions such as sulphur and mercury were also satisfactorily preserved in specially developed tubes. The tubes were preserved without rupture also on compression of the substances listed below. Far-reaching hydrolysis on shock compression of nickel fluoride hydrate resulted in the evolution of a considerable amount of free water and hydrogen fluoride; phosphorus oxide nitride decomposed almost completely into nitrogen and phosphorus and layer phosphorus oxides, and copper sulphate partly decomposed to free copper.

Systematic studies have been made to develop cylindrical preservation tubes which would permit the shock compression of solid–condensed gas mixtures in order to carry out various chemical interactions.[32] The task of preserving condensed gases after their compression in SW involves considerable difficulties. Firstly, metals become brittle at low temperatures, which increases the probability of the rupture of the tubes. Secondly, the high compressibility of condensed gases leads to considerable deformations of the tubes on compression and decompression which leads to the rupture of the tube under the conditions of increased brittleness of the material. Thirdly, even if the liquefied gas does not vaporise during compression, the effect of high residual temperatures after rarefaction causes its transition to the gaseous state, which creates high pressures within the tube capable of rupturing it. However, special tube designs, providing for the removal of the excess gases, made it possible to preserve the solid product. As a result of the investigations carried out, the interactions of various substances with liquid nitrogen and solid carbon dioxide have been achieved. In particular, the possibility of the formation of niobium nitride and carbide on shock compression of a mixture of powdered niobium with liquid nitrogen and carbon dioxide has been demonstrated.[32] The interaction of various metals with hydrogen yielded hydrogen-containing titanium and manganese compounds as well as other substances. It has been shown that saturated hydrocarbons and water can be used as sources of hydrogen in such interactions.[33]

In order to obtain CO_2 in the solid state under normal conditions, attempts have been made to oxidise SiC under the conditions of shock compression.[34] Mixtures of powdered SiC with gaseous or liquid oxygen were subjected to shock compression in preservation tubes. The study of the compression products by X-ray diffraction and infrared spectroscopy showed that the interaction of oxygen with SiC involves the oxidation of the latter and the formation of a non-molecular lattice structure. Heating of oxidised SiC to 800–1300 K removes carbon oxides. The cylindrical tube can be improved; in order to simplify the pattern of wave interactions and to eliminate the head wave, it is sufficient to insert a metallic rod along the axis of the tube. In this case the specimen in the form of a cylindrical layer is subjected to shock compression. This design can be used to achieve structural transformations[35] and also in technological processes—for the pressing of tubes.

Thus the generation of very high pressures, temperatures, and macroplastic deformations of specimens in cylindrical preservation tubes has a positive influence on many physicochemical processes. However, these conditions and the complex pattern of wave interactions in the test specimens restrict the scope of studies of the behaviour of substances under dynamic compression, whose class is limited mainly to metals and refractory compounds.

III. PLANAR DYNAMIC COMPRESSION

The creation of devices for the preservation of substances subjected to compression by planar SW ensured the possibility of dynamic compression at a lower maximum pressure but with a simpler pattern of wave interactions than in the cylindrical scheme. Planar compression also has the advantage over compression by converging waves because the SW parameters are susceptible to measurement and the compression conditions are controllable.

1. Experimental Technique

Fig. 4 illustrates schematically the designs for the preservation of substances after shock compression using a planar compression scheme. The shock waves in the tube and in the specimen are generated by a planar detonation wave of an explosive charge in contact with the tube (Fig. 4*a*) or as a result of the impact of a metallic

plate accelerated by the detonation product (Fig.4*b*).[33] After an appropriate selection of experimental conditions, the shock compression pressures in the test substance and in the material of the tube are equalised by repeated reflections of the SW from the bottom and lid of the tube when the latter is made of a material whose dynamic rigidity is greater than that of the test substance.[33] The scheme in Fig.4*b* is more convenient because impact by plates generates SW with a rectangular profile. A change in the thickness of the impact plate makes it possible to measure or calculate more rigorously the residence time of the test substance in the compression phase. In order to obtain more information, several specimens of different test substances were placed in a planar cassette (Fig.5*b*), which was in its turn inserted in the tube (Fig.5*a*), which made it possible to study the behaviour of different substances in a single experiment under comparable conditions. Such formulation of the experiment permits in addition the variation of the number of reflections of the SW in the specimen by using different thicknesses of the latter (Fig.5*c*).

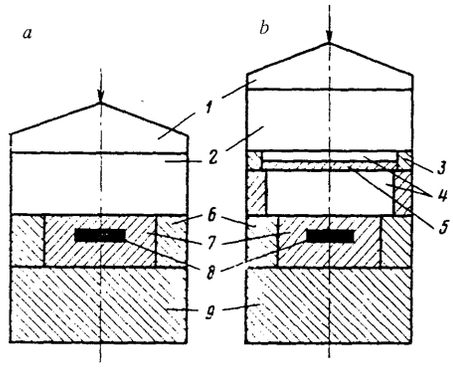


Figure 4. Schematic illustration of devices for planar shock compression: *1*) generator of planar detonation wave; *2*) explosive charge; *3*) metallic ring; *4*) air gaps; *5*) impactor; *6*) protective ring; *7*) tube; *8*) test substance; *9*) metallic support.

The pressures developed in the specimens depend on the dynamic rigidity of the material of the tube and the compression conditions and can range from 1 to 150 GPa.[33]

Despite the simpler compression pattern of the test substance when planar SW are used compared with the cylindrical compression scheme, the determination of the characteristics of the substance as a function of pressure requires a larger number of independent experiments with preservation of the specimen, also the compression conditions can be different in different experiments. A method has therefore been developed whereby it is possible to achieve in a single experiment the shock compression of one or several different substances over a wide range of fixed pressures with subsequent preservation of the specimens for physicochemical analyses.[36] Under the conditions created in this method, as the SW is propagated in a metallic block, the maximum pressure and temperature fall sharply after the wave has travelled a relatively short distance. A calibration relation between the pressure and the thickness of the metallic layer has been determined with the aid of special measurements.

The shock wave was generated in a copper block by the impact of an aluminium plate 1 mm thick accelerated by the products of a planar detonation of a thin layer of explosive; the distance traversed by the impactor was 2.5 cm. (We may note that at 45.5 GPa the maximum temperature of the copper reached 670 K, while the residual temperature T_{res} (Fig.1) was 480 K; at 13.2 GPa the corresponding temperatures were 380 and 330 K respectively.) The metallic block was then replaced by a set of plates of the required thickness, between which thin specimens of the test substance were placed. They were subjected to repeated shock compression at different fixed pressures.

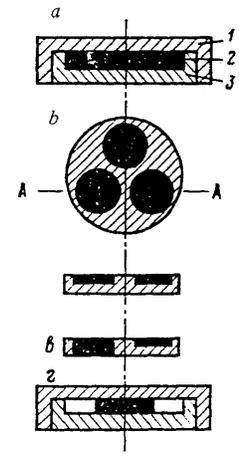


Figure 5. Design of the preservation tubes: *a*) preservation tube: *1*) lid; *2*) test substance; *3*) bottom of tube; *b*) cassette with three specimens which can be inserted in the tube instead of specimen *2*; *c*) section through the cassette along the AA line; *d*) preservation tube permitting a specified degree of deformation of the specimen as a result of its movement into the free volume on compression.

Comparison of the data obtained for the compression of the test substance in a layered container of this kind, where the shock compression times are short, with data obtained in independent experiments at the same pressures but for specified and longer compression times made it possible to obtain the dependence of the physicochemical characteristics on the shock compression time. In the experiment described the duration of the compression phase was determined from the instant of the appearance of the rarefaction wave arriving from the direction of the impactor; the pressure decreased over a period of 1.5–2.0 μs.

The specimens of the organic substances investigated, in the form of discs up to 0.7 mm thick at 15 mm in diameter or in the form of plates 50–100 μm thick, were placed within sockets in copper plates or were positioned between plates. In each layer several specimens of different substances could be placed. By selecting copper plates of a suitable thickness, it is possible to obtain up to 15 maximum pressures in the SW in the range from 10 to 40 GPa for several test substances in a single experiment. Since the thicknesses of the specimens are small, one can assume that there is no significant distortion of the wave front. The final pressure in the specimen after repeated compression is close to the

pressure in the copper plate, since the time of a single pass of the SW through the specimen is ~0.02 μs.

It is noteworthy that the contact of the thin layer of the test substance, which has been more compressed and heated to a higher temperature, with the metal heated to a lower temperature promotes the rapid removal of the residual heat from the specimen, ensuring a kind of quenching. Estimates have shown that, when a layer container is used for the shock compression of specimens of organic substances ~100 μm thick, the time required to equalise the temperature between the specimen and the metal is ~0.35 s. This design permits experiments at an initial temperature of 77 K, which improves the quenching conditions.

The planar compression scheme was used to study various substances and processes on shock compression. It proved to be most useful in the investigation of substances with a low thermal stability, such as, for example, organic substances.

2. Chemical Reactions of Organic Substances

Studies on the chemical reactions in organic substances which have undergone shock compression were initiated at the Institute of Chemical Physics of the USSR Academy of Sciences in 1965. The experiments performed using planar compression schemes revealed,[37] and this was subsequently confirmed in relation to many monomers, that exposure to a shock wave induces rapid polymerisation.[38] Polymerisation was observed for different classes of monomers which usually polymerise via radical (acrylamide) or ionic (trioxan) mechanisms. In addition, many monomers (tolane) which are extremely difficult to polymerise under the usual conditions do polymerise in the SW. The investigations showed that polymer chains are formed when the amplitude of the SW reaches a specific value, which differs for different monomers. The yield of the polymer formed and its average molecular weight initially increase with the SW amplitude and then diminish owing to the thermal degradation of the polymers. With increase of pressure, not only the length of the macromolecules changes but there is also a possibility of a change in the structure of the polymer chain. It has been shown[38] that these chemical reactions under the influence of SW cannot be reduced to a trivial thermal process and fusion or to a combination of static compression and heating.

The possibility of the formation of macromolecules as a result of polycondensation has also been considered. It has been demonstrated that the action of a SW in a mixture of α-aminoacids with silica gel can entail the formation of polypeptide molecules.[39] The authors[39] attribute a dehydrating role to silica gel, which displaces the reaction equilibrium towards the formation of polypeptides.

The first studies on the effect of SW on polymer systems (butadiene–nitrile, butadiene–styrene, natural and other raw rubbers) showed that in this case there is a possibility of the cross-linking of the macromolecules with formation of a three-dimensional network and their partial degradation.[40,41] The study of polymers with saturated bonds, such as poly(metyl methacrylate), polypropylene, polyethylene, and polystyrene showed that, regardless of the initial supermolecular structure (spherolitic, fibrillar, and quenched structureless) the effect of the SW leads to the formation of an isotypical fine spherolitic structure.[42] The use of a layer container permitted the study of the structural changes in polymer films at pressures differing by only 1.0–1.5 GPa in the range 10–40 GPa.[43] It was shown that the transition to the fine spherolitic structure takes place at pressures of 13.5, 23, 25, and 27 GPa for polybutylene, polypropylene, polyacrylonitrile, and Lavsan respectively. The degree of deformation of the specimens was estimated from the relation $\varepsilon(\%) = 100(R - R_0)/R_0$, where R_0 and R are the radii of the specimen before and after shock compression: it did not exceed 1%. Comminution of the supermolecular formations and an increase of the microhardness in the surface layers have been noted in a study[44] of the shock compression to 10 GPa of crystalline polycaprolactam and amorphous poly(methyl methacrylate).

A more detailed analysis of the above results was carried out in other investigations.[45,46] The efforts of many investigators, mainly Soviet scientists, were subsequently directed towards a more detailed study of the physicochemical properties of organic substances subjected to shock compression in preservation tubes and towards the elucidation of the mechanisms of the chemical reactions which occurred.

Three fields of study have now arisen. Firstly, we have the investigation of the reactions of low-molecular-weight organic substances, which include isomerisation, for example, the conversion of *trans*-stilbene into *cis*-stilbene,[47] condensation—the formation of polypeptides from α-aminoacids,[39] the dimerisation of polynuclear compounds such as anthracene, phenanthrene, and pyrene,[48] intramolecular rearrangements, for example, the formation of poly-β-alanine together with the usual vinyl polymerisation of acrylamide,[49] and finally the degradation reactions which partly accompany the processes enumerated above.

The data on the degradation of aromatic and cyclic compounds by SW proved to be the most informative.[50,51] It has been shown that shock compression of benzene disrupts the benzene ring and leads to the formation of a considerable amount of ethane and hydrogen, greatly exceeding the amount of the degradation products formed on compression of the less thermally stable hexane and cyclohexane. The authors of these investigations suggest that the disruption of the molecular structure is of a non-thermal character and that a change in the valence angles in the rigid benzene ring is impossible without the breakdown of the latter. On the other hand, aliphatic groups in cyclohexane can undergo a change in valence angles with subsequent relaxation. It has been concluded that the decomposition of benzene molecules takes place directly in the SW front where the most intense microplastic deformations take place.

Presumably the formation of defects, active centres, etc. in the SW front constitutes the "initiating" stage of the subsequent reactions. The isomerisation and condensation reactions can also be considered from this standpoint.

The second field of study involves the investigation of polymerisation reactions and demonstration of the activating role of the SW front in this process. The initiating role of the SW front in the polymerisation of acrylamide (AA) under the conditions of shock compression was first noted by Adadurov et al.[52] The experiments were performed at 77 K and a pressure where the polymer is not yet formed. Next, when not more than 30 s had elapsed since the experiment, the tube was cooled with liquid nitrogen, opened at this temperature, and the physicochemical analyses of the specimens were begun also at 77 K. The presence of active polymerisation centres at a concentration of 10^{20}–10^{21} kg^{-1} in the compressed AA specimens was confirmed by EPR. The SW front was regarded as a generator of active centres. The subsequent rise in the temperature of the SW-activated specimens led to an explosive AA polymerisation process, which was investigated calorimetrically. The data obtained and calculations led to the conclusion[52] that, depending on the temperature conditions established in the compression stage and obtaining after the removal of pressure, polymerisation can take place

either as a slow after-effect or as a thermal explosion. At high shock compression pressures, accompanied by the development of high concentrations of active centres and high temperatures, the polymer can be formed in the compression stage during a period shorter than 10^{-6} s. The presence of active polymerisation centres in AA compressed by a SW of brief duration has been confirmed by EPR.[53] The polymers were formed on subsequent dissolution of the specimen in tetrahydrofuran.

γ-Irradiation of the majority of solid monomers at low temperatures is known to lead to the formation of stable active centres which initiate the polymerisation process on subsequent temperature rise. A study has been made[54] in which AA specimens γ-irradiated in doses up to 6.3 Mrad were subjected to shock compression at 16 GPa and 77 K. It was shown that, for the dose indicated, the yield of the "purely radiation" polymer was 20%. The shock compression of non-irradiated AA led to the formation of 5% of the polymer, while that of the irradiated AA afforded 40% of the polymer. This yield exceeds by a factor of almost 2 the simple sum of the contributions by both influences and is almost an order of magnitude higher than the yield of the polymer due to the action of the SW alone. In the next Section of the review, in the analysis of new possibilities in dynamic investigations, direct proof of the decisive role of the SW front in the polymerisation of AA will be given.

We may note that the polymerisation mechanism under the conditions of shock compression described above is not the only one possible. Thus study of the polymerisation of trioxan[55] showed that the structural changes in the monomer during the compression stage are decisive for the formation of polyformaldehyde. The propagation of trioxan chains in microcrystalline and single crystal specimens has been investigated[56] as a function of the variable pressure and duration of the shock compression. It was concluded that the polymerisation takes place during shock compression and that the growth of polymer chains is virtually limited to this period. An increase of pressure entails an appreciable acceleration of the chain growth. The polymer chain propagation process is influenced significantly by the degree of perfection of the crystal lattice. Analysis of the changes in the characteristics of the products of the polymerisation of single-crystal trioxan under different conditions of compression by SW [57] showed that the molecules undergo a deformation which accelerates all the stages of the polymerisation process. Active centres are formed on partial degradation of the monomer molecules in the SW front, whereupon their number increases with increase in the degree of deformation.

The study of the transformations in polymers subjected to shock compression can be regarded as the third field of study. The observed degradation and cross-linking reactions[40,41] can also be accounted for from the standpoint described above in connection with the consideration of the mechanism of the polymerisation reaction. It is noteworthy that the long chains in the polymer macromolecules should be more sensitive to deformations in the SW front and the breaks in these chains should entail the formation of radicals. The subsequent reactions of these radicals can lead either to the degradation of the macromolecules or to their cross-linking with formation of a three-dimensional structure depending on the characteristics of the polymer, the presence of double bonds in the chain, etc.

In the study of the changes of the supermolecular structures of polymer specimens subjected to shock compression, the radiothermoluminescence (RTL) method was resorted to in addition to optical microscopy and X-ray diffraction.[43] Polypropylene (PP), in which any initial structure is converted into a fine spherolytic structure at a threshold pressure of 23 GPa, has been studied in greatest detail. It has been shown by the RTL method that, at a pressure below the threshold value the crystal structure breaks down and the fraction of the amorphous phase increases. It has been suggested that the compression in the SW front induces the formation of defects in the polymer structure and the annealing of defects or the rearrangement of the supermolecular structure at the elevated residual temperatures increases the fraction of the amorphous phase. The low-temperature luminescence peak observed by the RTL method for PP specimens compressed at 77 K indicates the possibility of the breakdown of the supermolecular structures. This low-temperature transition can be due to the local relaxation of the ends of the macromolecular chains along the boundaries of the cracks formed and on the surfaces of the lamellae. It has been suggested that the structural changes take place in polymers "activated" by the SW front immediately after the removal of pressure owing to the effects of the residual heat and can only be interpreted when account is taken of all the factors accompanying shock compression.

Studies of the chemistry of the shock compression of organic substances in which the observed effects were also explained have been published in recent years.[58-60] The conclusions reached are close to our ideas described above and reduce to the fact that shock compression can induce chemical reactions, including unusual reactions, which cannot occur for equivalent combinations of static pressures and temperatures and that the reactions can take place in the SW front itself. It has been suggested that the "anomalous" effects can be the result of "catastrophic shock".[58] According to this concept, the shock waves generate a high concentration of defects and cause the mechanical rupture of chemical bonds and the relative displacement of neighbouring groups in the SW front. As in the present author's investigations discussed above, the author[58] states that much more attention should be devoted to the possible processes in the SW front, such as the generation of a non-equilibrium state and defects, the breaks in chemical bonds, etc.

IV. THE REALISATION OF SPECIFIED THERMODYNAMIC CONDITIONS IN THE DYNAMIC COMPRESSION OF CONDENSED SUBSTANCES

The interrelation between the pressure in the SW front and the temperature developed there, limits the possibility of achieving sufficiently high pressures without the accompanying marked heating of the test substance. Shock compression of a precooled substance also does not lead to an appreciable reduction of this temperature. In other words, the region of the $P-T$ diagram immediately adjoining the P axis is in principle inaccessible in experiments with SW.

The attainment of this region under the conditions of dynamic compression at pressures up to 100 GPa and above is essential for the study of the metallation of dielectrics, including condensed gases, for the reduction of residual temperatures after intense dynamic compression, and for the prevention of the annealing of the high-pressure phases formed and the decomposition of the newly formed chemical compounds, etc. Despite certain advances in the technique of high static pressures, at the present time pressures in excess of 50 GPa can be obtained only on compression of extremely small amounts of test substances. Under dynamic conditions, the above restrictions are not limiting.

1. Stepwise Compression and Dynamic Isentropic Compression

The possibility, in principle, of a decrease in the temperature rise of the test substance on shock compression by organising a stepwise compression of the specimen by a series of successive SW of lower amplitude is known from the SW theory. For the same final pressure, the final temperature of the specimen is reduced in stepwise compression and the degree of compression of the test substance increases (because of the decrease of the thermal component of the pressure). As a result, the states located on the shock adiabat for repeated stepwise compression should approach the states on the compression curve at the absolute zero. Experiments in which shock adiabats have been recorded with double shock compression of certain substances have been described in the literature. An X-ray diffraction method for the investigation of regular regimes involving the oblique reflection and oblique collision of SW as well as the head on collision of SW introduced to achieve the double compression of magnesium, aluminium, perspex, paraffin and water have been described.[61] It has been shown that double compression states correspond to comparatively low temperatures and small thermal components of the pressure. For example, double shock compression of aluminium to a density of 1.57 g cm^{-3} results in the heating of the specimen to ~1100 K; on the other hand, in a single compression to the same density, the temperature reached is ~3800 K. The thermal components of the pressure in single and double compressions reach 20 and 6 GPa respectively.

P–V diagrams have been determined[62] for marble subjected to double shock compression. The shock wave emerging from marble was reflected from a copper plate with a high dynamic rigidity and was propagated through the marble specimen already subjected to compression by the first wave. It has been shown that at pressures in excess of 15 GPa the state arising in double compression significantly differs from the state in single compression and approach the "cold" compression curve.

It is seen from the examples presented that double shock compression makes it possible to reduce the temperature developed in the test substance. Repeated SW compression or "smooth" dynamic compression should make the process even closer to an isentropic process. Repeated SW compression of water and boron nitride to pressures of 100 GPa has been carried out[63] in order to measure their electrical conductivities. The compression of test specimens placed between two copper plates was achieved by creating shock counterwaves of identical amplitude, which were generated by the synchronous collision of aluminium plates accelerated by the explosion products. It was shown in these experiments that in the region of 80 GPa boron nitride exists in a semiconducting state. The electrical conductivity of water is ionic and at a pressure of 30 GPa the specific conductance reaches a plateau as a result of the attainment of the total dissociation of water molecules.

The dynamic isentropic compression (DIS) of fused quartz and liquid hydrogen to ~500 GPa has been achieved[64] with the aid of an explosive magnetic flux compression technique. It was shown that, up to pressures of 500 GPa, quartz does not become an electrical conductor. At 200 GPa liquid hydrogen has a density of ~1 g cm^{-3} and at this pressure its transition to a metallic state was not observed. The density of hydrogen subjected to repeated shock compression has been measured[65] by the γ-ray diffraction method; the density was determined by measuring the diameter of a cavity converging towards the centre of a cylindrical shell (accelerated by detonation products) in which hydrogen was present initially in the gaseous state. It was shown that, at pressures in excess of 280 GPa, the density of hydrogen increases from 0.8 to 1.3 g cm^{-3}, which the authors suggest is due to the transition of hydrogen to the metallic state.

Several studies postulating various schemes (not realised experimentally hitherto) for the achievement of the isentropic compression of a substance under dynamic conditions have been published. Thus Yampol'skii,[66] who also carried out a general analysis of the state of the problem, suggested that the "smooth" compression of the specimen be effected by the products of the detonation of a charge located at some distance from the target; the period of increasing pressure then depends on the size of the gap. It has also been suggested that media with variable dynamic rigidities be employed. In the propagation of SW through such media there is a possibility of the transformation of the SW into a wave with a smooth increase of pressure in the front.[66] A gas-dynamic method for the low-temperature compression of solid hydrogen placed in a copper cooling conductor has been proposed.[67] The isentropic compression of hydrogen can be achieved in this method by transmitting pressure to the cooling conductor via a gas which is compressed by a metallic cylinder moving towards the centre as a result of being acted upon by a descending detonation wave.

All the proposed methods have been designed to record the low-temperature states of substances in the course of shock compression or DIS and do not provide for the preservation of the specimens of the test substances. In the above methods the test specimens break down after compression and fly apart in the form of a plasma, particles, fragments, etc. One should also note the difficulty and high cost of creating a cylindrical converging detonation wave and reproducible strong magnetic fields.[68]

2. The Generation of Low-Temperature States of Condensed Substances in Dynamic Compression in Experiments with Preservation of the Specimens

Systematic studies designed to generate experimental conditions ensuring low-temperature dynamic compression were initiated in 1970 at the Institute of Chemical Physics of the USSR Academy of Sciences. Attention was concentrated on the development of simple schemes for the compression of substances placed in preservation tubes and ensuring a closer approach to the isentropic regime compared with the stepwise shock compression.

Since the temperature on the isentropic curve corresponding to the given degree of compression is linearly related to the initial temperature of the test substance, it follows that, in order to be able to carry out experiments at lower temperatures, it was necessary to develop also designs for preservation tubes permitting experiments with the specimens cooled to 4.2 K. Evidently dynamic isentropic compression at low temperatures should lead to the appearance of states of the test substance which are located in the immediate vicinity of the compression curve at the absolute zero. Estimates have shown,[66] that, for example, in the shock compression of lead to 100 GPa, the maximum temperature reaches ~9200 K, while the residual temperature is 1800 K. Isentropic compression to the same pressure of the specimen initially at room temperature should heat lead by not more than ~900 K and from 4.2 K only up to 12 K.

We shall consider briefly below the principal experimental schemes developed for the achievement of low-temperature dynamic compression and shall discuss the differences in the states of various substances whose specimens were

preserved after repeated shock compression and after DIS.[33,69-73]

Analysis of the propagation of SW in media with variable density indicates the possibility of their transform ion into smooth isentropic compression waves.[63,73] Since it is very difficult to prepare a medium with a continuously varying medium density, systems consisting of layers of substances having different dynamic rigidities, which made it possible to reduce significantly the amplitude of the first SW and to increase the number of subsequent waves in the repeated SW compression of the test specimen, have been used.[69,70] Organic substances having low thermal stabilities were employed as a kind of thermoindicators.

In one of the methods proposed, a decrease of the amplitude of the first SW was achieved by placing a buffer system of plates (screens) made up of different materials in front of the specimen.[69] In these experiments it was possible to reduce the amplitude of the first SW in acrylamide and polypropylene to ~3 GPa for a final pressure of 50 GPa. In the case of repeated shock compression for the same pressure in the absence of screens, the amplitude of the first wave was ~13 GPa and for the specimen initially at room temperature AA underwent total thermal degradation and PP underwent a thermal structural transformation. On the other hand, under conditions close to DIS, i.e. in the presence of screens, there was only partial degradation of AA and the structural transformation of PP did not take place.

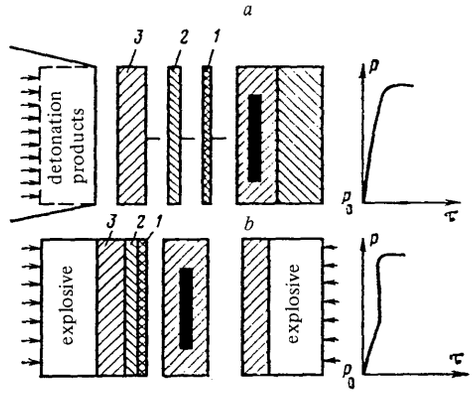


Figure 6. Schematic illustration of devices ensuring the dynamic isentropic compression (*a*) and successive compression of the test substance by the isentropic wave and then the shock wave (*b*); the three layer impactor is made up of perspex (*1*), aluminium (*2*), and copper (*3*) plates. The time variation of the pressure in the specimen during compression is shown on the right.

The compression scheme illustrated in Fig.6, which permits the achievement of "smooth" compression of the test substances placed in preservation tubes by successive impacts against the tube by plates (impactors) with increasing dynamic rigidity, proved to be the most rational.[33,70] By a planar detonation of an AA charge, SW are generated and propagated in this method over a system of plates, made, for example, of copper, aluminium, and perspex; when the shock wave reaches the free surface, it initiates at first the motion of the perspex plate, which acquires the highest velocity by virtue of its high compressibility. Impactors made of aluminium and copper require lower velocities and the velocity of the plate is lower the greater the dynamic hardness of its material. The final pressure in the target is generated after impact by the copper plate.

Qualitative analysis of this method leads to the conclusion that, when the distance between the layer impactor and the target is varied, it is possible to achieve a wide variety of different profiles of the compression waves accompanied by a longer period of increasing pressure at the given point in the specimen compared with the usual repeated SW compression. It is significant that, when the law governing the decay in the target (the lid of the tube) of the shock wave generated as a result of the impact by the first thin plate is known, it is possible to select the thicknesses of the materials in such a way that after the first impact the test substance is acted upon by a SW with a very low amplitude.

A series of experiments have been carried out[73] on the repeated shock compression and DIS of an organic monomer—acrylamide, using various compression schemes (Figs.4*b* and 6*a*), up to 40 GPa at initial temperatures of 293, 77, and 10 K. After the experiments, the tubes were cooled to 77 K in order to fix the products formed, then opened at the same temperature, and the properties of the specimens were investigated also starting with 77 K. It was found that repeated shock compression of AA at 293 K led to its complete degradation or at 77 K it gave rise to a 30% yield of the polymer. On raising the temperature of the cooled specimens, postpolymerisation was not achieved, apparently as a consequence of the annealing of the active centres. In the course of compression at 10 K, 10% of the polymer was formed and the concentration of active centres remained the same, so that, on raising the temperature of the specimen to 293 K, vigorous polymerisation took place; the final yield of the polymer reached 40%.

Under DIS conditions at 293 K and 77 K, 20 and 3% of the polymer were formed respectively; at 10 K the yield of the polymer was virtually zero. On raising the temperature, polymerisation did not occur apparently owing to the inadequate number of active centres, the possibility of the appearance of which was reduced probably as a consequence of the lack of high pressure gradients in the SW front. Degradation was not observed over the entire experimental range. The results obtained indicate a significant decrease of temperature in DIS compared with repeated shock compression.

The development of methods for the DIS of substances at low initial temperatures in experiments with preservation of the specimen created a possibility for the investigation of frozen gases or systems in which they are included. Tube designs were developed which permit the preservation of the above specimens after their dynamic isentropic compression to 50 GPa. Carbon dioxide, chlorine, water, a mixture of water and carbon dioxide, and a mixture of n-heptane and chlorine were investigated.[33] In order to observe the changes in the state of the substances at low temperatures, the RTL method was used. It was found that the RTL spectra of many substances subjected to compression differ from the initial spectra. For example, after the compression of CO_2, new peaks appear on the luminescence curve. The $C_7H_{16} + Cl_2$ mixture subjected to DIS loses its capacity for a rapid reaction in the region of the phase transition (at about 153 K), which is characteristic of an untreated mixture of these reactants.

The demonstration of the possibility of carrying out experiments with condensed gases under dynamic compression conditions with preservation of the specimens and of the

direct observation of their properties must be regarded as the principal result of studies of this kind carried out hitherto.

As research expands, an increasing number of processes which are favourably influenced by low-temperature dynamic compression are being discovered. For example, the low-temperature dynamic compression to 20 GPa of trioxan subjected to preliminary γ-irradiation induces a much greater increase in the yield of the polycrystalline polymer and of the number of chains that might have been produced by the simple summation of the compression and irradiation effects.[74] In order to increase the yield of the dense boron nitride phase, two-layer cylindrical tubes made of different materials have been used,[75] which led to an increase of the multiplicity of the shock compressions and to a decrease of the temperature developed in the specimen. When the molybdenum–iron pair was used, it was possible to attain a 70% yield on compression of boron nitride without additives. A layer of liquid hydrogen, which served simultaneously as the cooling agent, was used[76] as a light pressure transmitting substance. It was shown that, in the zone where DIS is active in copper, brass, and nickel specimens, a significant decrease of microhardness and of the density of traces of deformation was observed compared with the corresponding characteristics in the region of shock compression. The profiles of the compression waves in DIS are being investigated[77,78] and various schemes for the practical employment of layer systems have been proposed.[79]

We should like to draw attention to the following possibility which appears in connection with the development of methods for the attainment of DIS. In DIS the microshear deformations at the front and the temperatures developed are significantly reduced even by comparison with repeated shock compression, whereas the duration of action, the character of the decrease of pressure, and the degree of macroplastic deformations of the tubes with the specimen are virtually the same in both cases. DIS by itself does not as yet allow the separation of the role of microshear deformations and of the temperature factor, since a weakening of the influence of the former induces also a decrease of the temperature of the substance behind the front. However, preliminary heating of the substance under DIS conditions makes it possible to obtain $P-T$ conditions identical with those in the usual repeated shock compression and hence to isolate the influence of the SW front in a "pure form".

Two processes of different kinds, occurring on shock compression, have been investigated: the structural transformation of boron nitride into a dense diamond-like modification[80] and polymerisation of AA.[33] It has been shown that, when the experiments are carried out at low temperatures or at room temperature, the replacement of repeated SW compression by DIS prevents the structural transition in boron nitride. On the other hand, preliminary heating above 400 K is sufficient for the dense form of boron nitride to be produced both on DIS in 70% yield and on repeated shock compression. It has been shown in relation to the polymerisation of AA that the SW front is in fact responsible for the process, because preliminary heating of AA specimens up to the melting point did not increase the yield of the polymer, which remained an order of magnitude lower on DIS than on repeated shock compression.

3. The Attainment of Ultrahigh Pressures as a Result of the Head-on Collision of Shock Waves

The collision of planar SW and the attainment of ultrahigh pressures in specimens and their subsequent preservation for analysis have been achieved.[81] This became possible as a result of the construction of a symmetrical apparatus (Fig.7) in which two specimens of the test substance are subjected to two identical initial SW directed towards one another. We may note that modern explosive experimental technique makes it possible to synchronise with high accuracy the detonation of two identical explosive charges which accelerate identical impactors. An important requirement is the displacement of the test substance within the apparatus into the region not subjected to negative pressures which arise as a result of the interaction of the rarefaction counterwaves. When the layer 5 is made of a very dense material, for example, tungsten, it can promote a considerable increase of pressure in the reflected wave. If the shock adiabats for the materials and the velocities of the impactors are known, then the maximum pressure in the test substance can be estimated beforehand.

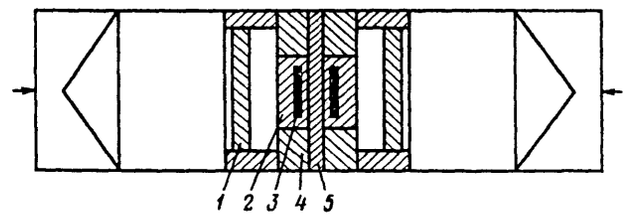


Figure 7. Schematic illustration of the experimental apparatus for the attainment of ultrahigh pressures on collision of planar shock waves: *1*) impactor (may be a multilayer impactor); *2*) preservation tube; *3*) test substance; *4*) protective ring; *5*) layer of a hard material.

The use of the method described for the treatment of substances by dynamic pressures makes it possible to increase greatly the maximum attainable pressures without complicating the existing systems and devices designed for the creation of high pressures in a one-sided impact. For example, at the velocities of the copper impactors usually employed, amounting to $\sim 2.0 \times 10^3$ and 2.5×10^3 m s^{-1}, pressures of ~50 and ~65 GPa respectively are developed in copper (or steel). The head-on collision of such impactors generates pressures of ~120 and ~170 GPa. The use of powerful explosives and greater charges makes it possible to increase the velocities of copper impactors to $\sim 3.5 \times 10^3$ m s^{-1}, whereupon pressures up to ~300 GPa can be achieved in a head-on collision.

Experiments have demonstrated[81] that the usual tubes with test substances withstand effectively pressures up to ~100 GPa. Above this pressure, changes in design are necessary in order to eliminate considerable macroplastic deformations. Experiments on the dynamic compression of polyethylene (PE) and polystyrene (PS) at pressures up to ~120 GPa have been carried out.[81] In order to achieve DIS, the head-on collision of two compound impactors made of perspex, aluminium, and copper has been used. Copper impactors, which determined the final pressure, had a velocity of $\sim 2 \times 10^3$ m s^{-1}. Analysis of the PE and PS specimens preserved after compression showed that they remained monolithic and semitransparent and were free from traces of degradation. A gel fraction was formed in PS in 100% yield owing to the formation of cross links, but, judging from the infrared spectrum, the decomposition of the benzene ring was not observed. We may note that

repeated shock compression of PS to 50 GPa leads to only partial formation of the gel fraction. It is known that γ-irradiation of polystyrene with a dose of 100 Mrad is necessary for the formation of 30% of the gel fraction.

The methods examined and the results show that both shock and isentropic compression waves or their various combinations can be used in head-on collisions. For example, a specimen subjected to preliminary DIS can be then acted upon by SW (Fig. 6*b*). The possibilities of this method have already been described.[33] In other words, it is now possible to achieve a controlled dynamic compression of specimens of different kinds and also to follow the variation of the state of the substance both in the course of compressing, by employing high-speed recording apparatus, and directly during the analysis of the preserved specimens.

V. CHEMICAL SYNTHESES AND THE PREPARATION OF ULTRAHARD MATERIALS DIRECTLY IN THE DETONATION WAVE

Until recently dynamic methods for the preparation of ultrahard materials (UHM) such as diamonds and the dense modifications of boron nitride were based solely on the use of SW.[82] The initial materials are placed in flat or cylindrical preservation tubes which are penetrated by SW generated by a detonating explosive placed on the outside. Thus the detonation of explosives is used indirectly as a mixed external source of energy for the generation of SW. It has been shown in recent years that the detonation of charges made of a mixture of explosives and materials to be transformed (carbon or graphite-like boron nitride) entails the formation of UHM.[82] For this purpose, free mixed charges were detonated in stationary repeatedly reuseable hollow hermetically sealed containers, which made it possible to avoid the employment of disposable preservation tubes. It is significant that the detonation parameters (pressures up to 50 GPa and temperatures up to 6000 K) correspond to the region of the synthesis of UHM on static presses and overlap the part of the phase diagram which is difficult to attain, in which carbon and boron nitride exist in the liquid state.

The detonation synthesis[82] makes it possible to obtain diamond from various modifications of carbon, including amorphous modifications, with different fractional compositions. Depending on the experimental conditions, it is possible to synthesise the cubic modification or a mixture of the cubic and hexagonal modifications of diamond. Detonation diamond is a highly dispersed material with a high specific surface (20–150 $m^2\ g^{-1}$) and small coherent scattering regions (60–200 Å).

It has been shown that dense modifications of boron nitride can be obtained by detonation synthesis from both crystalline graphite-like and high-temperature boron nitride. Furthermore, depending on the detonation parameters of mixed charges, it is possible to achieve the specific synthesis of wurtzite-like or cubic modifications of boron nitride. As in the case of diamond, the particle size of the dense modifications of boron nitride is small—ranging from 0.05 to 5 μm, while the density is significantly lower than its theoretical value: 3.15–3.30 $g\ cm^{-3}$.

In considering the possible mechanisms of the formation of UHM, the hypothesis that carbon and boron nitride melt with subsequent crystallisation of the UHM from the melt appears to be of greatest interest. In particular, this hypothesis is supported by the synthesis of diamond from amorphous forms of carbon.

The question of the possibility of the synthesis of oxides, nitrides, and carbides directly in the detonation wave has been examined in a review.[33] The examples quoted indicate extensive new possibilities for the dynamic methods for acting on matter.

VI. THE ROLE OF PLASTIC DEFORMATION IN DYNAMIC EXPERIMENTS AND THE POSSIBILITY OF COMPARING THE RESULTS OF DYNAMIC AND STATIC INFLUENCES

In discussing the questions concerning the role of plastic deformations, it is essential to differentiate two types of information obtained in the dynamic compression of matter. These include various characteristics recorded in the course of shock compression in the central region of the specimen, which is not yet affected by rarefaction waves, and the data obtained from them, for example, compressibility, electrical properties, etc. When the specimen is compressed by a planar shock wave, its central region is subjected to a one-dimensional compression without transverse deformations. The compression of a substance by powerful SW, whose amplitude greatly exceeds the yield point, makes it possible to neglect the effect associated with the hardness of solids. One can therefore assume that compression to a given pressure is the same as that which would have been achieved under a hydrostatic pressure of the same magnitude. This hypothesis is confirmed by the fact that in most cases the characteristics obtained by the shock compression methods merge smoothly, on extrapolation, with the characteristics obtained by static methods at lower pressures. For a number of substances, a satisfactory agreement has been observed as regards the onset of reactions in static and single shock compressions.[83,84] This applies, for example, to substances such as Fe, Bi, Si, Ge, P, I, Sb, Se, FeC, KCl, CdS, and GaAs.

It has been suggested that the martensite mechanism of the transformation predominates on compression by SW, whereas the change in the lattice is based on the cooperative displacement of many atoms over short distances. Such a transformation, which does not require an activation energy, can take place at very low temperatures at sonic rates of propagation, so that the particles of the new phase, with micron dimensions, are formed in the course of 10^{-7}–10^{-8} s. It has been suggested[84] that a complete identity of the phase transition pressures is possible for coordination crystals and layer structures with few defects. However, highly defective layer structures require higher pressures on compression by a SW than under static compression.

The specific features of shock compression are determined by the SW front in which the strongest microplastic deformations take place simultaneously with a sharp increase of the pressure and temperature. Despite the fact that the duration of the action of high pressure is only a fraction of a microsecond, i.e. is shorter by a factor of 10^7 than in static compression, there is sufficient time for the occurrence of a wide variety of physicochemical processes in the test substances. The acceleration of processes in the SW has been compared by many investigators with the results obtained at high static pressures in combination with shear deformation (HP + SD)—see the relevant review.[85] However, the difficulty of comparing these two methods consists in the fact that under static conditions one investigates as a rule P–T diagrams, while under dynamic conditions one obtains relatively simply and with a high accuracy P–V diagrams. On the other hand, the temperature can be in the main estimated only by calculation subject to various simplifying assumptions. Furthermore, the pressure range in dynamic

experiments greatly exceeds that in static experiments. The question of the possibility of a comparison has been analysed in greatest detail in the review of Dremin and Breusov.[86]

Information about the influence of the rarefaction waves on chemical and phase transformations of a given substance in the course of shock compression is virtually lacking in the literature. Certain considerations concerning the role of rarefaction waves in the formation of active polymerisation centres and their contribution to the residual temperature have been published.[33]

When questions associated with the comparison of the changes in the properties of substances preserved in tubes after shock compression or after DIS are discussed, the situation is different. As already mentioned, in this case the specimen is acted upon by the full phase of the SW, including the rarefaction waves. Under these conditions, the specimen in the preservation tube inevitably undergoes major plastic deformations which depend on the compression conditions and the formulation of the experiment.

In considering the experiments in which specimens were compressed in cylindrical tubes, attention was drawn above to the complex pattern of the wave interactions and to the occurrence of the intense uncontrollable plastic deformations of the substance, leading to additional heating of the specimen. Nevertheless, it is sometimes possible to carry out a qualitative comparison of the results of static and dynamic experiments. We shall consider this in relation to the formation of polymeric molecules from unsaturated carboxylic acids—maleic (MA) and fumaric (FA), which are *cis*- and *trans*-isomers. Under the usual conditions, these acids do not polymerise owing to steric hindrance. However, it has been shown[87] that under HP + SD conditions at 6 GPa they undergo oligomerisation. The oligomers have a molecular weight of 700–800, which corresponded to a chain consisting of 6–7 molecules.

These objects behave differently on shock compression. Whereas both oligomers with a chain length of 5–7 units and polymeric products with a molecular weight (MW) of 3×10^4 (chain length of 250 units) are formed in MA on compression in flat and cylindrical tubes, in FA polymeric products are detected only under the conditions of enhanced deformations in the cylindrical tubes.

When special precautions are taken, the use of a planar compression scheme makes it possible to alter specifically and within wide limits the degree of radial macroplastic deformations of the specimens. Fig.5 illustrates the design of tubes in the usual formulation of the experiment (*a*) and in an experiment where the degree of deformation is specified beforehand (*d*), in which the specimen is capable of being deformed during the period of the operation of the high pressure ($\sim 10^{-5}$ s) and of filling the free volume in the tube. Such conditions were first used in the study of the influence of the degree of macroplastic deformations as a function of pressure on the polymerisation of acrylamide in a layered tube.[88] An increase of the degree of deformation of the specimens from 1 to 17% at the same pressure led to an appreciable increase of the number of polymer chains formed and of the yield of the polymer.

The scheme for the creation of specified macroplastic deformations has been used also in the study of the possible abiogenic syntheses of aminoacids in nature from nitrogen-containing organic compounds using as an example the reactions of the ammonium salts of unsaturated carboxylic acids under dynamic compression[89] and on their deformation under the conditions of a high static pressure up to 6 GPa.[90] The dynamic compression of the specimens was achieved in five different regimes. Shock compression, shock compression with a specified degree of deformation (up to 160%), DIS by the usual method, and DIS with a specified degree of deformation of the specimens were achieved in the planar arrangement, while in the cylindrical version the system was also subjected to shock compression. The pressure was varied from 5 to 11 GPa and the initial temperatures were varied from 77 to 300 K.

It has been shown[89,90] that, both in the HD + SD method and on dynamic compression, the ammonium salts of acrylic, methacrylic, crotonic, fumaric, and maleic acids can be converted into aminoacids. The yield of aminoacids on dynamic compression depends on the pressure, the initial temperature, and especially on the compression regime. The maximum yield has been attained in the presence of enhanced macroplastic deformations (Fig.5d) in planar compression under DIS conditions and in the cylindrical compression scheme.

It is to be hoped that the proposed schemes for compression with a specified degree of deformation will make it possible to compare quantitatively the results obtained in the HP + SD method and in different dynamic compression regimes and to discover the role of the SW front, plastic deformations, kinetic factors, etc. in various physicochemical processes.

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The technique of dynamic experiments in which provision is made for the preservation of the test specimens after compression in order to investigate the changes which they have undergone made it possible to extend the range of thermodynamic parameters compared with the traditional studies of the state of substances in the course of shock compression with subsequent breakdown of the specimen. Treatment of specimens in the pressure range 10–100 GPa and above and at temperatures up to 10^4 K has become a realistic possibility. The useful volume of the specimens of the test substances is not limited at much higher pressures, in contrast to static apparatus.

The set of systems for the preservation of substances which have undergone dynamic (shock or isentropic) compression developed has made it possible to achieve various physicochemical transformations.

Methods permitting the attainment of controllable changes in the character of the dynamic compression and of the degree of deformation and also experiments at very low initial temperatures permitted in many instances the determination of the mechanism of the action of the SW and the role of its front. These experiments should promote the joint employment of the results of dynamic and static investigations, particularly in the HP + SD method. The possibilities of the dynamic method have not as yet been fully elucidated, as shown, for example, by the synthesis of ultrahard materials directly in the detonation wave.

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