High-pressure neutron scattering over the ages*

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Abstract
Trends in neutron studies at intermediate pressure are considered. They are
mostly associated with the broadening of the range of methods (elastic, inelastic,
small-angle scattering and depolarization of neutrons) and with the creation of
necessary equipment. It is shown that in the studies of pressure-induced and
stress-induced transitions, damping of tunnel modes due to elastic stress, and
similar problems, a combination of quasi-hydrostatic and purely hydrostatic
experimental conditions is essential. Tthe possibilities and perspectives for
new trends of studies at intermediate pressure are assessed.

1. Introduction

Problems which can be solved with the help of neutrons at high pressures are quite various,
and are closely connected to the phase transitions in matter during the change of volume. They
are, for instance, the nature of bonding in hydrogen-containing substances, the stability of
molecules and molecular ions, the stability of magnetic structures with competing types of
interaction, the nature of systems with intermediate valence and superconducting systems, and
many others. The behaviour at high pressure enables us to understand the structure, excitation
spectra and the nature of bonding in crystals at ambient pressure better.

A change of the specific volume is accompanied by changes of atomic and electronic
structure, interatomic distances, excitation spectra and physical properties. Various methods
of volume alteration are known. Irradiation (defect doping), and the addition of interstitial
atoms and molecules ('chemical' expansion) lead to $\Delta V > 0$; the substitution of big atoms
of a matrix with smaller ones ('chemical' pressure) and finally high pressures (hydrostatic,
quasi-hydrostatic and uniaxial) lead to $\Delta V < 0$. In addition, the volume can be changed
in thin films by the influence of the substrate and by electric and magnetic fields on account
of striction. Similar phenomena take place in the wide pressure range, but for soft materials
they occur in the kilobar range, and for hard materials they occur in the megabar range. The
intermediate pressure range is important because in this range various regimes of pressure

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* In memory of Professor J M Besson and Professor J Rossat-Mignod.

application are possible: hydrostatic, quasi-hydrostatic and uniaxial regimes; but at higher pressures the hydrostatic regime is impossible.

Neutron investigations at high pressure became a self-contained and perspective field quite recently, after the Abingdon Conference (1992) initiated by Professors R Nelmes and J M Besson. The International Seminars on Neutron Scattering at High Pressure held in Dubna (1994, 1999) and International Workshops MPa4 in Grenoble naturally continued the discussion of problems, which had been started in Abingdon, and revealed new tendencies in the use of neutron methods at high pressures [1, 2]. The main difficulty which limits the pressure range in neutron studies is the necessity to use unusually small quantities of sample, and this requires high luminosity and low background instruments and sufficient beam time. The best advantages for broadening the pressure range came from the use of the anvil technique. Progress in experimental facilities has promoted the application of different neutron techniques at high pressure for the solution of various physical problems.

2. Some history

Neutron scattering at high pressure has a long history. Because of the outstanding advantages of neutron scattering over other methods in specific cases, attempts had already been made even at the early stages of these studies [3]. An important step was made in the work [4] where a sintered alumina pressure cell with pressure range up to 30–35 kbar was introduced, and with the help of such a type of cell many experiments in various laboratories were carried out. This year the 30th anniversary of this work takes place, so it seems reasonable to make a brief overview of neutron studies at high pressure in the last three decades (1974–2004). In the first decade (1974–1983) the majority of experiments were done with piston–cylinder cells, studying the equations of state and phase transitions at rather low pressure with the help of elastic neutron scattering. Examples of such work are studies on Bi and its alloys (BiVI at ILL, BiIII, BiIV, BiVII at RRC KI together with ISSPh [5]), which were done with a Ti–Zr press at $T = 300$ K and $P = 30$ kbar. A number of various devices for different temperature ranges and various cells were designed, including one made of several superalloy single crystals. The principal disadvantage of these devices was the limited range of pressure.

In the second decade (1984–1993), while the luminosity of neutron diffractometers increased due to the use of multidetector data acquisition systems [6], the first experiments with the diamond anvil cell [7] were carried out, thus providing the extensive growth of pressure range up to 300 kbar for single crystals and up to 370 kbar for powder samples at the 5 MW reactor [8–12]. The main weakness of these experiments consisted in the very small amount of sample (less than 0.1 mm^3) and so the class of problems and methods was restricted to samples with only the highest scattering power and diffraction. Besides that, these experiments were very expensive because of the high cost of the anvils. For elimination of these shortcomings two ways were used. Cheap sapphire anvils were used in the pressure range up to 70–80 (a record 120) kbar for samples of volume about 1 mm^3 [13, 14]. For samples of volume 20–100 mm^3 and pressures up to 300 kbar a new type of special press [15–21] was created (the Paris–Edinburgh project under the initiative of Professors Besson and Nelmes). Both these ways were used in the various centres and ensured expansion of the class of problems and neutron methods used.

In the last decade (1994–2004), dedicated instruments for neutron studies at high pressure with dedicated beam time were created in JINR (Dubna) for thermal neutrons [22–25] and LLB (Saclay) for cold neutrons [26, 27] (with the support of Professor J Rossat-Mignod). The first experiments on the investigation of dispersion curves and density of states were carried out in anvil cells [28, 29] with inelastic neutron scattering, and pressure-induced phase
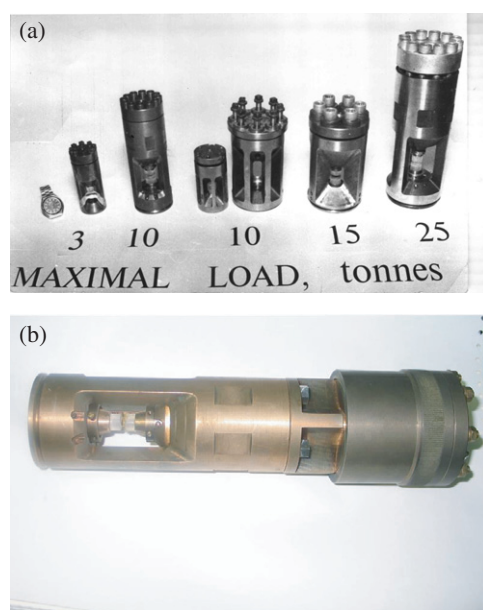
Figure 1. (a) Pressure cells from RRC KI (1993–1996) for low (Cu–Be) and high (steel) temperatures for elastic and inelastic neutron scattering with sapphire and diamond anvils. (b) Pressure cell from RRC KI (1997) for SANS and depolarization with sapphire anvils and pneumatic system. The pressure is measured before and after the experiment by the ruby fluorescence technique, and in the course of the neutron experiment by a gas manometer.

(This figure is in colour only in the electronic version)

transitions were studied with neutron depolarization and small-angle scattering [30]. At the Saclay instrument the pioneering high-pressure investigations of magnetics were carried out with strong stationary magnetic fields and low temperatures [31], and at the Dubna instrument structure, phase transitions and phonon and libron density of states in ammonium halides were investigated [32]. In this work the pressure range for investigations was broadened up to about ∼500 kbar for elastic scattering, up to ∼200 kbar for coherent inelastic scattering, up to 100 kbar for incoherent inelastic scattering and 80 kbar for neutron depolarization. So during the last decades we could see a significant progress of neutron investigations at high pressures in the extension of the pressure range, in the use of a wide ensemble of methods, in the construction of equipment for the measurement of pressure and loading (figures 1(a), (b)) (for example, the pneumatic camera with continuous loading and measurement for SANS and depolarization [30]), in the new types of anvils (moissannite [33], almazot and others), and in the new materials and pressure-conducting media. Extending the hydrostatic pressure range up to 90 kbar by capsulating at ISIS [34] is of great importance. Despite this progress, investigations in the intermediate-pressure range remain relevant, because only in this range is a comparison of measurements in hydrostatic and quasi-hydrostatic regimes possible for revealing the influence of defects, stresses and pressure inhomogeneity on phase transitions.

Let us consider examples of such influence.

3. Hydrogen tunnelling modes in α-Mn suppressed by elastic stresses [35]

Recent experiments [36–38] on the neutron diffraction and inelastic neutron scattering (INS) from solid solutions of hydrogen in α-Mn revealed the effect of hydrogen tunnelling at relatively
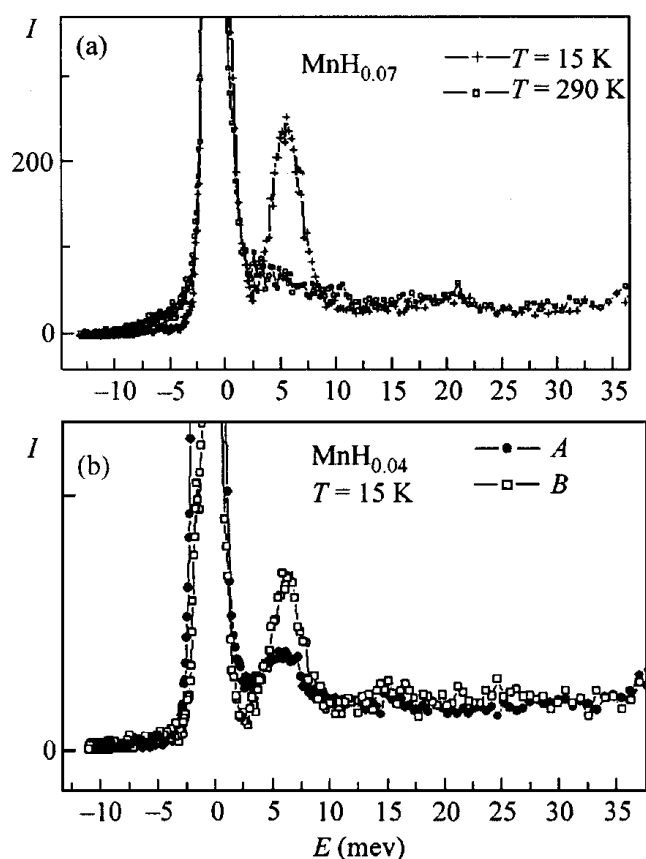
Figure 2. INS spectra measured on the DN-12 spectrometer at JINR, Dubna, (a) for $MnH_{0.07}$ at different temperatures and (b) for freshly prepared and triturated $MnH_{0.04}$ samples.

high temperatures (up to about 90 K). The effect was manifested by the appearance of a temperature-dependent intense narrow peak at 6.3 meV in the INS spectra, which was explained by splitting of the ground vibration state as a result of the tunnelling of hydrogen atoms between adjacent closely spaced 12e-positions. The energy of the observed tunnelling modes was about 30 times that for the other quantum systems [39, 40].

It was natural to suggest that a decrease in volume, leading to a reduction of the distances between possible positions of hydrogen atoms and to a change in the shape of the double-well potential, may significantly influence the tunnelling of hydrogen. In connection with this, we studied the effect of high pressures on the effect of hydrogen tunnelling in α-Mn by the method of incoherent INS.

The INS spectra of freshly prepared samples (figure 2(a)) showed that a decrease in temperature below 100 K leads to the appearance of a sharp resonance peak at 6.4 meV, which agrees with the previous results [38] and corresponds to the tunnelling motion of hydrogen atoms in the double-well potential. The peak amplitude significantly decreases upon dry or wet (ethanol) grinding of the samples (figure 2(b)). Upon loading in a quasi-hydrostatic mode in sapphire anvils, the INS spectrum exhibits a dramatic change: the peak at 6.4 meV disappears already at a pressure of about 8 kbar and is not restored either at higher pressures (17 kbar) or upon unloading (figure 3(a)). Since the phenomenon is observed in both $MnH_{0.04}$
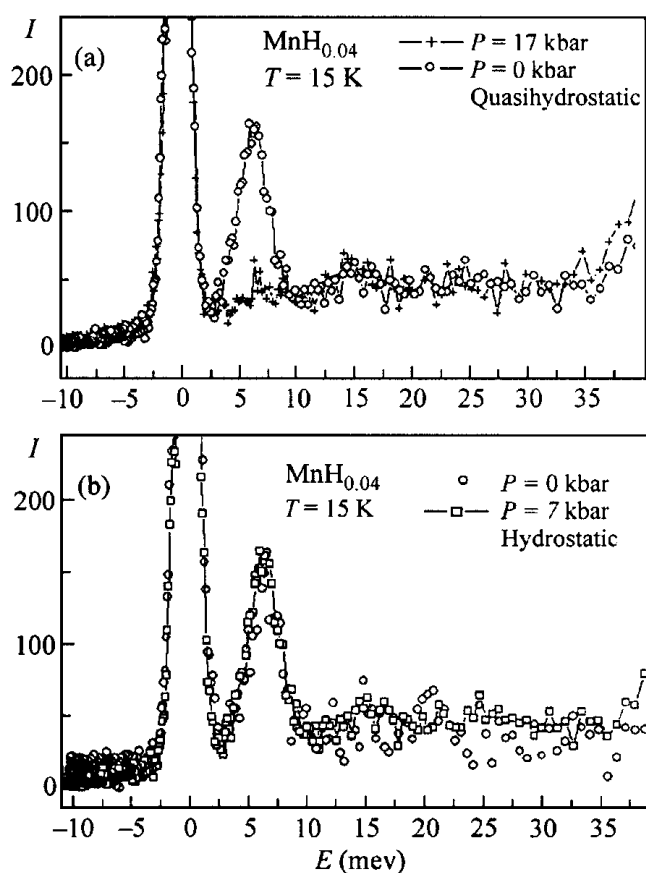
Figure 3. INS spectra measured on $MnH_{0.04}$ samples loaded with different pressures in sapphire anvils under (a) quasi-hydrostatic and (b) purely hydrostatic conditions.

and $MnH_{0.07}$ samples, the effect of pressure is independent of (or weakly dependent on) the hydrogen concentration.

The above results suggest that the effect of suppression of the hydrogen tunnelling states is related to the residual elastic stresses (or defects) arising in the samples as a result of nonhydrostatic loading or grinding, while the contribution of homogeneous compression is small. In order to verify this assumption, we conducted experiments under hydrostatic conditions, using freshly prepared samples loaded in anvils filled with Fluorinert. In this case, no significant changes in the INS peak at 6.4 meV were observed: the intensity and the width and position of the peak remained virtually the same upon hydrostatic loading (figure 3(b)).

Thus, changes in intensity of the INS peak related to hydrogen tunnelling are caused by the local elastic stresses in the sample, either induced by inhomogeneous pressure or produced by grinding, rather than by applied pressure as such.

The probability of tunnelling in a defect-free crystal is determined as

$$W \approx \Delta^2/\omega,$$

where Δ is the tunnelling band width and ω is the characteristic frequency of the dynamic displacements caused by the interaction with lattice vibrations [41, 42]. The probability of tunnelling in a crystal strained by elastic stresses must be significantly decreased by a shift δ

between the energy levels:

$$W \approx \Delta^2 \omega / (\omega^2 + \delta^2).$$

In the absence of local stresses, a shift of levels arises due to the dynamic displacements and determines the temperature dependence of the tunnelling probability. In our experiments, the shift of levels is determined by static (rather than dynamic) displacements. The large magnitude of this shift is explained by the elastic moduli of the metal matrix being much higher as compared to those of a quantum crystal [43]. Analogous manifestations of the tunnelling modes suppressed by local elastic stresses should probably be observed in other quantum systems as well, albeit being less pronounced in cases of lower elastic moduli.

4. Pressure-induced and stress-induced orientation transitions in magnetic systems [44]

It was established by the method of neutron depolarization that in MnAs the phase transition to the high-pressure ferromagnetic phase is observed. The phase exists in the range from 20 to 90 kbar. It was shown by the neutron diffraction method that in this phase the spin-reorientation transition from the ferromagnetic orthorhombic phase to a new magnetic phase takes place when the temperature decreases. The new phase has both the ferromagnetic and antiferromagnetic components of a magnetic moment. The transition causes the deviation of magnetic moments in the (011) plane by the angle ~50° and decreases the ferromagnetic moment in comparison with the high-temperature ferromagnetic phase. At the same time, the structure parameters of the Mn and As atoms are not changed with decreasing temperature. The only exception is the x parameter of the Mn atoms, whose value slightly increases. The x parameter increasing must change the magnetic interaction between the nearest and next-nearest neighbours and can be a factor 'provoking' the magnetic phase transition.

The changes in magnetic structure of the iron borate $FeBO_3$ [45, 46] were studied by the neutron diffraction method under hydrostatic and quasi-hydrostatic pressure up to 40 kbar. Previously we established that at the pressure 17 kbar in $FeBO_3$ the spin-reoriented phase transition 'easy plane–easy axis' occurs. This causes the change of the angle between the directions of iron magnetic moments and the $\langle 111 \rangle$ crystallographic axis. But this transition was not observed in the later studies of $FeBO_3$ single crystals under hydrostatic pressure by the Mössbauer spectroscopy method using synchrotron radiation, and the Mössbauer data came into conflict with the neutron data. Because the stress distribution inhomogeneity in a sample can influence the spin-reorientation transition parameters the neutron experiments were performed on powder samples in sapphire anvils in the case of both pure hydrostatic (in a liquid) and quasi-hydrostatic pressure. It was shown that the spin reorientation takes place in the quasi-hydrostatic conditions at pressure higher than 14 kbar, but in hydrostatic conditions it does not occur up to the pressure of crystallization of the liquid (21 kbar). The obtained results can be explained by the effect of 'switching off' the external pressure in the rhombohedral weak ferromagnets predicted by Ozhogin and Farzetdinova [47]. The effect consists in the fact that in the case of applying an external pressure along the rhombohedral axis, as in the Mössbauer experiments, the magnetic system is not susceptible to the external pressure, but in the case of its applying in the orthogonal direction, the pressure of the spin-reorientation transition is minimal (6 kbar for $FeBO_3$). In the neutron experiments in the quasi-hydrostatic conditions on powder samples an intermediate case takes place. Hence the performed studies show that the spin-reorientation transitions in magnetic systems under high pressure can be divided into two types: pressure-induced transitions (as in MnAs) and stress-induced transitions (as in $FeBO_3$ and Fe_2O_3 [48]). The first ones involve volume decrease and change of interatomic distances. The second ones are caused by the values and directions of elastic stresses in the crystal.

5. Excitations at high pressure [$\omega(q)$ and $g(\omega)$]

Structural phase transitions in crystals can be related to the selective sensitivity of the phonon modes to variation of thermodynamics parameters of temperature and pressure. Thus the temperature development of structural instability in crystals is well known. That is why for an understanding of the nature of structural transitions at high pressure the basic phonon characteristics, the density of states $g(\omega)$ and dispersion curves $\omega(q)$ should be investigated. It is very important in this case that, unlike the optical methods for studying excitations at phase transitions in crystals, the neutron methods are more comprehensive because they cover the whole region of phase space. This can be illustrated by the study of the pressure-induced orientation transitions in ammonium halides and the high-pressure study of pyrolytic graphite.

5.1. Behaviour of phonon and libron modes in ammonium halides

The influence of high pressure on the libration mode of ammonium ions and the phase-transition-induced changes on the mode's behaviour are among the most intriguing aspects of the dynamics of ammonium halides [49]. The variation of structure and the dynamics of ammonium halides NH_4X (X = F, Cl, Br, I) under high pressure up to 100 kbar have been described using elastic and inelastic neutron scattering data [50–54].

The equations of state and the pressure dependence of interatomic distances were determined in ammonium halides. It was established that at compression the distance between the ammonium cation ND_4^+ and the anion X^- (X = Cl, Br, I) significantly decreases. In doing so the covalent bond length l_{N-D} of the ammonium ion almost does not change with pressure increase, and is equal to 1.02(2) Å at any halide anion radii. It was found that the phase transition between the disordered CsCl-type cubic phase II and ordered phase IV and V in ammonium halides could be ascribed to a critical value for the deuterium positional parameter u_{cr} ($u = l_{N-D}/a\sqrt{3}$, where l_{N-D} and a are the N–D bond length and the lattice parameter, respectively). For the pressure range where the condition $u < u_{cr}$ is satisfied, ammonium halides exist in phase II, and for $u > u_{cr}$, we have the parallel ordered (Cl, Br) phase IV or antiparallel ordered (I) phase V.

The influence of high pressure on the rotational potential barrier in ammonium halide phases was investigated. It was found that the phase transitions from the disordered cubic phase to the phases with different ammonium ion ordering result in an increase of the rotational potential barrier value figure 4. This is matched by distortion of the model symmetric two-wells potential which is changed into asymmetric form by increasing the depth of one potential well. The potential change is a result of ammonium ion ordering.

The vibration spectra of ammonium halide NH_4I were studied under pressure up to 100 kbar by incoherent inelastic neutron scattering. The behaviour of the L and TO frequencies gives evidence for the appearance of interaction between them, i.e., for the libron–phonon hybridization. This effect (figures 5,6) results in the successive attraction and repulsion of the vibration branches and, respectively, in sizeable changes of the Grüneisen parameters. The nature of this phenomenon is, likely, similar to the hybridization of other types of excitations: in particular, magnons and phonons in magnetic crystals. With each ammonium ion there can be associated an orientation vector directed from the centre of the NH_4^+ tetrahedron to one of its vertices. The librations are caused by the oscillations of the orientation vectors about their equilibrium positions, just as the atomic magnetic moments oscillate in magnons. However, there are no spatial correlations between the librations of neighbouring NH_4^+ ions, so this branch is virtually dispersionless.
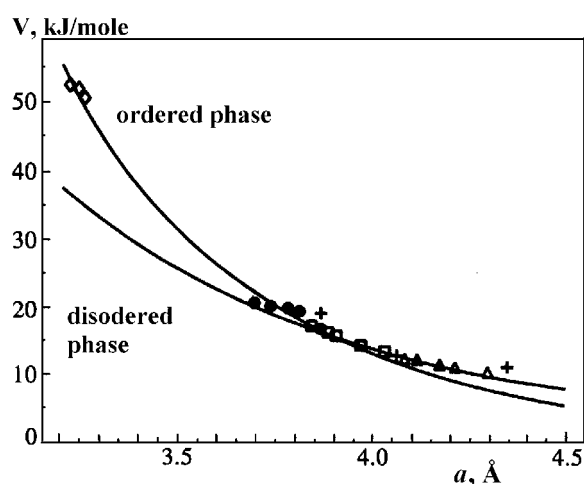
Figure 4. Rotation potential barrier as a function of the lattice parameter for ordered and disordered phases in ammonium halides: △—NH_4I, ☐—NH_4Br, ●—NH_4Cl, ◇—NH_4F, +—optical data. Solid lines are the theoretical curves for $V_0 = B/a^c$, $c = 5$ for disordered phases, $c = 7$ for ordered phases.

Thus the simple models of interatomic potential change at pressure-induced phase transitions are used to describe the observed structural changes and they provide possibilities to predict the structural behaviour of these systems for higher pressure.

5.2. Changes of phonon dispersion in graphite at high pressure

The lattice dynamics of graphite at high pressures was studied by inelastic scattering of neutrons for the first time using the anvils technique [55, 56]. The pressure dependence of frequencies for hexagonal-axis-polarized phonons in strongly anisotropic layered crystal lattice of graphite has been measured up to 60 kbar. It is shown that the pressure effect, resulting in a contraction of interlayer distances, gives rise to a monotonic hardening of the measured frequencies. The investigated longitudinal acoustic branch does not change its sinusoidal shape under pressure, while the continuous evolution from quasi-two-dimensional to three-dimensional behaviour is found for a transverse acoustic branch (figures 7, 8). It is pointed out that the observed higher rate of pressure variations of the lattice dynamics parameters as compared to the structural anisotropy can reflect changes of the crystal potential in graphite related to the existing high-pressure phase transformation from the layered to more isotropic lattice (figure 9).

6. Problems for the future

Among the great number of problems for which the use of intermediate pressure range is relevant, several problems can be accentuated for investigations in the next ten years.

(a) *Study of metastable systems at high gas pressure.* In recent studies it was shown that the implantation of hydrogen in the lattice of a metal at high pressure (2–50 kbar) and temperature (from ambient to 1000 K) can give rise to a number of new phenomena. They are: formation of metals with superabundant vacancies [57], formation of hydrogen-induced ordered or decayed structures in the initially disordered alloys [58, 59], and hydrogen-induced amorphization [60]. Neutron experiments at high pressure and
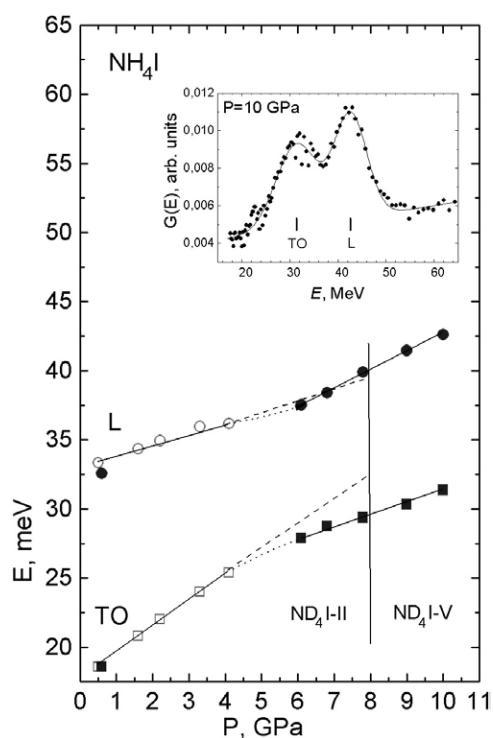
Figure 5. Pressure dependence of the L and TO modes of NH_4I. Solid lines are the linear interpolations of experimental data in the ranges 0–4 and 6–10 GPa, respectively; dotted lines are the interpolation in the range 4–6 GPa; and dashed lines are the linear interpolation to the II–VI phase-transition point in ND_4I. Black circles and squares are the data from [53]; white circles and squares are the neutron data from [52]. Inset: generalized density of vibration states in NH_4I at 10 GPa. The peaks are approximated by Gaussians and the background by a straight line.
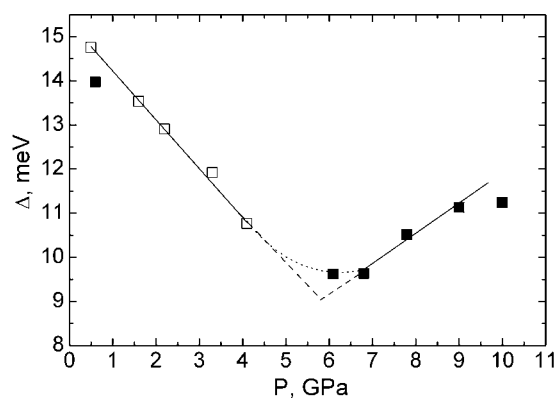

Figure 6. Pressure dependence of the difference between the frequencies of the L and TO modes. Black squares are the data from [53] and white squares are the data from [52]. In the ranges 0–4 and 7–10 GPa the experimental data are interpolated by linear functions.

temperature *in situ* will be able to clarify the mechanism of these transformations and the possibility of creation of new metallic materials after hydrogen desorption.
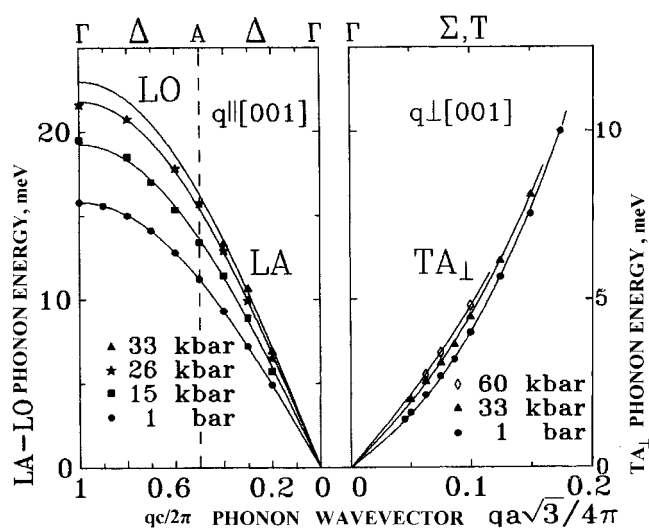
Figure 7. Phonon dispersion curves in graphite at different pressures. The LA–LO branch at 15 kbar is taken from [28]. Solid lines correspond to the parameters of the best fits of the measured frequencies to the relations described in [56]. A part of the investigated pressure values is presented.
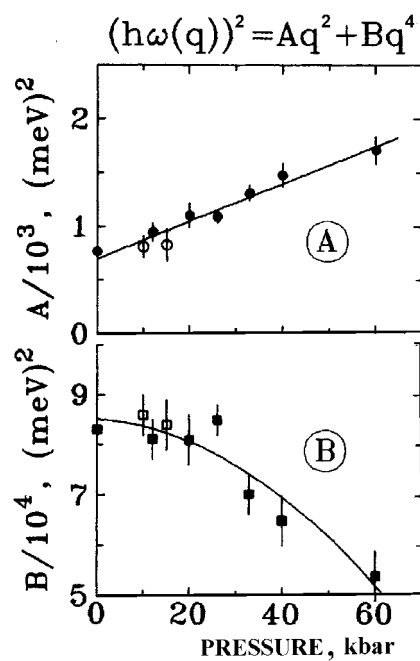

Figure 8. Pressure dependence of parameters A and B in the dispersion law [56] for the $TA_\perp$ phonon branch. Open symbols are determined from the data of [28]. The solid line represents the best linear fit to the relation $A = A_0(1 + \delta P)$ (with $A_0 = 0.70 \times 10^3$ meV^2 and $\delta = 0.025$ kbar^{-1}).

(b) *Pressure and irradiation. Polyamorphism.* It was shown in recent years that decrease of volume at high pressures gives rise to new modifications in amorphous and disordered
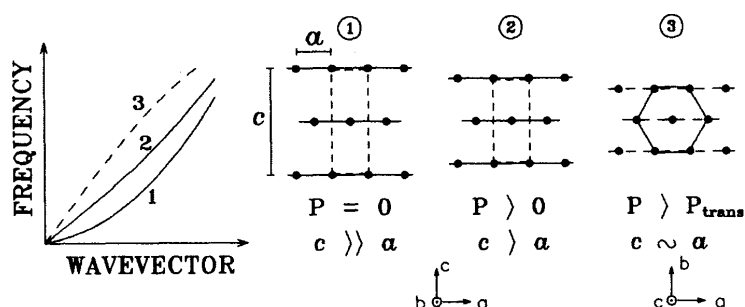
Figure 9. Schematic drawings of changes in the phonon spectrum of a layered compound when its structure anisotropy decreases (from '1' to '2'). Drawings labelled with '3' correspond to a crystal structure and phonon dispersion after a possible phase transition. It is shown that the higher rate of the interlayer spacing variation as compared to the intralayer one may result in 90°-reorientation of the main symmetry axis under the phase transition, as happens in graphite [66]; this can be a common property of some layered lattices.

systems (light and heavy amorphous ice [21, 61, 62], change of radial distribution functions in glasses [63]); this phenomenon was named polyamorphism. Recently we observed a similar phenomenon in irradiated diamond: it was the transition from diamond-like glass to graphite-like glass [64]. Whereas pressure leads to the increase of density, the irradiation (which is doping by defects) leads to its decrease. A study of the influence of both factors will ensure very big variation of density (up to 2–4 times), and study of various phase transitions under volume changes will be possible.

(c) *Excitations in the low dimensional systems at high pressure*. This problem, being studied insufficiently, seems the most suitable for the intermediate pressure range, because relatively big single crystals and McWhan-type pressure cells for hydrostatic conditions can be used. A study of the dispersion curves for phonon and magnon excitations along with the $\omega(q)$ peculiarities, connected with dimensionality and interactions of excitations, would enable the observation of transitions at changes of dimensionality and volume (1d–2d–3d) and accompanying changes of excitations, as was observed in pyro-graphite [56].

(d) *Pressure and perfect crystals*. An important and unexplored problem is the change of distribution of electron and spin density under change of volume. The most precise data can be obtained by the observation of dynamical scattering effects in perfect crystals (e.g. Pendellösung in semiconductors and magnetics [65]) at high pressure. These types of experiments have never been carried out so far; they require pure hydrostatic conditions and big size of sample. They can be done in piston–cylinder cells in the intermediate pressure range.

7. Conclusion

The intermediate pressure range makes possible a comparison of the results obtained in hydrostatic and quasi-hydrostatic conditions and under uniaxial compression, and allows studies of the effect of stress and defects on phase transitions, quantum diffusion and so on. This is important because at higher pressures hydrostatic conditions are impossible. In this pressure range almost all new and old neutron methods can be used, which are hardly applicable at higher pressures. In the next ten years we can expect the use of all neutron methods for experiments with volume changes first in the intermediate pressure range.

Acknowledgments

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