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Chapter 3.29

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XAFS and thermodynamic variables (pressure and temperature) to investigate matter

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The importance of exploring the physical properties and phase transitions of matter under high pressure and/or high temperature are illustrated using basic thermodynamic concepts. The specific role of the X-ray absorption fine structure (XAFS) is addressed with emphasis on its unique sensitivity to the atomic short-range order. Current techniques suitable for performing high-pressure and/or high-temperature XAFS measurements are discussed. Examples of applications to elemental systems as well as perspectives and trends in innovations are briefly reviewed.

1. Introduction

X-ray absorption spectroscopy (XAS) is an advanced experimental technique that is sensitive to changes in the local atomic and electronic structure. In particular, the X-ray absorption fine structure (XAFS) contains information on the local structural order around selected photoabsorbing atomic species in terms of short-range properties of the radial (and possible higher order) distribution function g(r). Moreover, low-lying unoccupied electronic states are also probed by XAS in the near-edge region. Modifications of the atomic and electronic structure can thus be observed and studied by XAFS under different thermodynamic conditions.

The environmental hydrostatic pressure P and temperature T are the most obvious, and externally tunable, parameters that are useful to explore the complex phase diagrams of condensed matter. Within the thermodynamic formalism, the equilibrium state of a homogeneous (P, V, T) system is defined by P and T, with the system volume V(T, P) being a unique function of these two variables in agreement with the equation of state f(P, V, T) = 0. A fundamental state function to characterize possible phases is the corresponding Gibbs free energy,

$$G(T, P) = U(T, V) - TS(T, V) + PV,$$
 (1)

where U(T, V) and S(T, V) are the internal energy and entropy of the system, respectively, and V = V(T, P). The evolution of the system in contact with an environment at constant (T, P) is determined by the condition $\Delta G \leq 0$. Correspondingly, the stable phases of a system are characterized by the lower possible values of the Gibbs free energy among different competing aggregation states. Clearly, the variation of one or both thermodynamic variables T and Pallows one to explore the phase diagram of the system and obtain information on the boundaries of the stability regions of the various phases through which phase transitions take place.

Thermodynamic systems may survive in metastable states that are characterized by a higher G than the stable phases.

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For instance, various material processing techniques (vapour deposition, high-pressure amorphization processes *etc.*) can be used to generate metastable disordered solid structures when the limited diffusion does not allow relaxation to the stable phase. Metastable undercooled liquids can also be obtained and studied for a variety of substances by rapid cooling of the melts.

The investigation of condensed matter in a wide (P, T) range around ambient conditions $(T \simeq 300 \text{ K}, P \simeq 0.1 \text{ MPa})$, determination of the phase boundaries and the measurement of several possible structural and electronic properties provide important information on the atomic interactions, improving fundamental knowledge and capabilities for prediction. The accessible (P, T) range usually includes so-called extreme thermodynamic conditions, to which the present chapter is largely devoted.

A typical *P* range to induce phase transitions in condensed phases extends up to 100 GPa and beyond, a range which is currently achievable using static high-pressure experimental devices. The temperature parameter *T* can be also varied over a wide range (typically from 10^0 to $2-3 \times 10^3$ K in static measurements using cryostats and furnaces or even into the 10^4 K range using laser heating). Further details of XAFS under high *P* and *T* conditions can be found in the relevant literature: see, for example, Itié *et al.* (2016), Di Cicco & Filipponi (2015), Abe *et al.* (2018) and references therein.

Knowledge of the fundamental physical properties of systems under various (P, T) conditions, including structural, dynamical and electronic properties, is of fundamental importance. X-ray (or neutron) diffraction is the standard technique for probing the structural properties of crystalline samples through Bragg reflections, and is also used to obtain information on the radial distribution function in disordered systems. However, as is also better explained in other chapters of this volume, XAFS provides useful complementary information to that obtained by diffraction, being extremely sensitive to the short-range structure around selected atomic species and to phase transitions occurring in condensed matter. Generally speaking, structural properties of matter under variable pressure conditions can be obtained by various experimental techniques, where X-ray (neutron) diffraction and XAFS provide accurate long-range and short-range (roughly within 5 Å) reconstruction of the atomic distribution functions, respectively.

XAFS measurements can be performed over a wide (P, T) range with a number of basic advantages over possible alternatives: (i) the substantial blindness to long-range crystalline order makes XAFS data of different phases relatively similar and directly comparable, (ii) the atomic selectivity of XAFS provides direct access to sample properties even using complex cells that are suitable for providing the required sample environment and usually contribute a smooth absorption background, and (iii) the element selectivity allows one to measure structural properties with sufficient sensitivity around minority atomic species in compounds and alloys and also in the diluted limit where all other structural techniques fail.

For all of the above reasons, the use of XAFS for the investigation of materials under variable and extreme (P, T) conditions is continuously increasing. In the following sections, we will briefly review some of the techniques used to obtain XAFS spectra under extreme conditions and a few selected examples of applications. This necessarily short overview is mainly confined to the direct experiences of the authors and cannot be exhaustive; it also relates to a rapidly growing field.

2. Typical experimental setups at XAFS beamlines

X-ray absorption spectroscopy beamlines using energy-scanning modes and transmission measurements are still the option of choice for the collection of spectra with the best signal-tonoise ratio and energy-resolution performance. This scanning mode is typically obtained by measuring the incoming and transmitted photon flux while rotating a double-crystal monochromator (for a selected Bragg reflection). For wide XAFS scans, the most stable instruments are those that exploit a continuous spectrum from a synchrotron-radiation source such as that generated by a bending magnet or a wiggler. For these beamlines smooth energy-dependent conditions are realized for a given flux-resolution trade-off, while X-ray mirrors are often used to improve focalization conditions and for harmonic rejection purposes together with possible crystal detuning. High-quality XAFS experiments in a wide (P, T)range (see Fig. 1) benefit from the occurrence of simple conditions with regard to (i) the full compatibility of the beamline with a sample environment (cell/devices) able to generate the required thermodynamic conditions and (ii) the availability of ancillary techniques and diagnostics for in situ sample characterization. In this chapter, we briefly describe some of the techniques that are suitable to perform XAFS measurements under nonstandard (P, T) conditions, referring to other contributions in this volume with regard to sample design, which is another fundamental requirement to obtain accurate and reliable XAFS measurements. In particular, a sample design and cell windows must be selected in order to



Figure 1

The regions of the (P, T) plane that are accessible by sample-environment devices that are typically available at XAFS beamlines.

access the elemental edges under consideration under different thermodynamic conditions. Generally speaking, the sample confinement using cell windows required by static high-pressure techniques limits the photon spectral range to hard X-rays (E > 7 keV), with exceptions related to special cell configurations and the use of X-ray Raman techniques. On the other hand, an extended spectral range can be obtained for XAFS experiments at variable temperatures under vacuum conditions.

XAFS energy-scanning beamlines are usually equipped with standard commercial temperature devices that can easily be adapted to perform XAFS experiments. The use of simple liquid-nitrogen cryostats or resistive heating furnaces can typically cover the 77–700 K range. Most beamlines are also equipped with helium cryostats allowing measurements down to 4–10 K.

XAFS experiments at higher temperatures are relatively easy to perform under high vacuum using special resistive heating furnaces (see, for example, Filipponi & Di Cicco, 1994) and the only concerns regard temperature homogeneity and possible sample reaction/contamination. Temperatures of up to about 3000 K can be reached and maintained for the typical timescales of XAS measurements. Further information on various high-temperature experiments, including levitation techniques that may possibly be used for contactless XAFS experiments, can be found in the review by Neuville *et al.* (2014).

Many beamlines can easily offer suitable devices for XAFS measurements under vacuum and, obviously, ambient pressure conditions. However, high-pressure conditions present considerable experimental challenges. We refer to the review by Shen & Wang (2014) for detailed information about high-pressure measurements using synchrotron radiation, including XAFS.

Accurate XAFS measurements at high pressure have similar experimental requirements and problems in common

with various experimental setups that are worth a brief discussion in this chapter. Ideal hydrostatic conditions require sample confinement in a pressure-transmitting medium in the presence of suitable X-ray windows that are part of the pressure device. The materials of both the windows and the pressure-transmitting medium contribute to the total X-ray absorption and may considerably reduce the useful flux, especially at low photon energies, possibly increasing the harmonic contamination of the X-ray beam. Thus, there is an important limitation regarding the use of X-ray windows. For example, single-crystal windows, such as those used in the diamond anvil cell (DAC) devices shown in Fig. 2, deviate part of the X-ray beam when the photon energy matches the required conditions for Bragg reflection. As a consequence, narrow absorption peaks are observed in transmission-mode XAFS spectra that are spoiled by the presence of these artefacts. Due to the small sample size (linear dimensions of 10-100 µm), DACs are mostly used on energy-dispersive beamlines and more recently also on energy-scanning beamlines using suitable X-ray focusing. Bragg reflections from DACs can be displaced out of the energy region of interest more easily on energy-dispersive beamlines, and therefore suitable experimental techniques based on polycrystalline confinement gasket materials are recommended, especially for energyscanning modes. Recent advances based on repeated measurements over a small angular range of DAC orientation (Hong et al., 2009) or the use of nanocrystalline diamonds and the possibility of extreme focusing on XAS beamlines are expected to overcome some of those drawbacks (see, for example, Nakamoto et al., 2011; Coppari et al., 2012). Temperature-dependent measurements using DACs can typically be carried out in the 10-1000 K range using suitable cryostats and heating devices. Diamond deterioration becomes important above 700 K, so a suitable inert or vacuum environment is necessary for high-temperature measurements, which are usually carried out using sample laser heating



Figure 2

From left to right: sketch of the diamond anvil cell (DAC) configuration for XAFS measurements in transmission mode. The DAC can be oriented to maximize the angular range for the collection of diffraction Bragg peaks (as well as to minimize spurious effects in XAFS transmission data). The external force is imposed on the tungsten carbide seats and then transmitted to the diamond anvils, reaching pressures up to the 100 GPa range, which are usually measured by an optical probe. The temperature can be increased using resistive heating devices (usually up to 1000 K) or by laser heating. The right-hand part of the figure shows a schematic drawing of the (bi-conical cylindrically symmetric) sample section of a large-volume Paris-Edinburgh-type (PE) press. The figure shows the boron–epoxy gasket, the graphite heater and its internal cavity filled with a sample, as well as the thermocouple and several small parts forming the sample environment. The grey arrows indicate the application of external forces to obtain high pressures in the sample region.

through the diamonds. Standard DACs are normally usable in the 8–15 keV photon energy range, although XAFS measurements can also be collected below 8 keV using special DAC configurations (drilled diamonds, beryllium gaskets).

High-pressure cells with sample volumes in the cubic millimetre range are an option of choice for XAFS experiments in transmission mode on energy-scanning beamlines. As an example, a suitable technique for high-pressure and high-temperature XAS measurements (typically up to 10 GPa and 1500 K) was developed as a modification of the original toroidal gasket technique (Khvostantsev et al., 1977) and exploits the Paris-Edinburgh press (Besson et al., 1992) with the use of double conical gaskets as first employed at LURE (Katayama et al., 1997) and successively further developed on BM29 at ESRF (Filipponi et al., 2000) and elsewhere. A typical sample assembly for high-pressure XAS measurements using these devices is shown in Fig. 2. Gaskets are usually manufactured from sintered rods of a boron-epoxy mixture offering sufficient strength and low X-ray absorption. The sample, which is often a mixture of the selected substance under investigation with other substances used as a pressure marker or pressure medium, is placed in a millimetre-sized internal cylindrical cavity surrounded by a thin graphite cylinder used for resistive heating. Pressure is applied along the vertical axis by two anvils with tungsten carbide insets. The low photon energy limit of this technique is typically 8-9 keV, which is achievable with small gaskets and focalized beams.

Experiments under extreme conditions also benefit from additional diagnostics and, in particular, independent probes of the (P, T) thermodynamic parameter are required for the various experimental techniques. Thermocouples, local pressure gauges (such as the ruby fluorescence method in DACs) and X-ray diffraction using equations of state have commonly been used to estimate the thermodynamic parameters under static conditions. Knowledge of the thermodynamic parameters is moreover not always sufficient to unambiguously establish the sample phase, in particular when metastable states occur. Crossing a phase-transition boundary by increasing or decreasing the pressure often results in a hysteresis loop owing to the slow crystallization kinetics of the new phase, especially at low temperature. An undercooled liquid state may persist well below the equilibrium melting temperature. A simultaneous diffraction check of the nature of the crystalline sample components, which is currently available at most XAFS beamlines, is always recommended (Filipponi et al., 2000). The XAFS phase sensitivity can also be exploited by monitoring the sample X-ray absorption at fixed energy on a significant spectral feature during continuous temperature scans (Filipponi et al., 1998), in which transition temperatures between the different phases and possible hysteresis loops can be revealed very effectively.

3. XAFS applications and perspectives

XAFS experiments at variable temperature using commercial heating/cooling devices began as soon as the XAFS technique became popular in the 1970s and their applications are too

numerous to be mentioned here. Specific high-temperature furnaces using suitable sample-environment designs were gradually introduced, allowing XAFS measurements to be performed at extremely high temperatures (Filipponi & Di Cicco, 1994; Filipponi *et al.*, 2000), such as, for example, liquid and solid rhodium up to about 2300 K (Di Cicco *et al.*, 1999).

The first published high-pressure experiment using XAFS was performed using a modified Bridgman anvil method (Ingalls et al., 1978), reaching 6.4 GPa on an FeS₂ solid sample. The potential to apply this technique to liquids was addressed. An evolution of the device to allow X-rays to pass along the anvil axis (diamond or B_4C) was later developed and applied to solid NaBr and germanium (Ingalls et al., 1980). Polycrystalline B₄C anvils are suitable for energy-scanning XAS experiments under extreme conditions, with an upper pressure limit of about 8-10 GPa. A wide-aperture high-pressure cell with 3 mm thick beryllium windows limited to 0.8 GPa with direct compression of the fluids has also been described and applied to RbCN (Werner et al., 1982), and many applications in the kilobar (0.1 GPa) range of pressures using different autoclave systems have been realized (see, for example, the experiments on liquid selenium by Tamura et al., 1995).

As mentioned above, the use of a conventional DAC for high-pressure XAFS studies is hampered by the presence of sharp Bragg reflections from the diamonds, which are seen as absorption peaks. A successful high-pressure technique for collecting XAFS spectra has been developed by the highpressure group at LURE (see the applications to phase transitions in GeO₂; Itié *et al.*, 1989; Itié, 1992) using dispersive XAFS. In this configuration, the entire XAFS spectrum is collected with a subsecond acquisition time, allowing an effective *in situ* alignment procedure aimed at displacing all Bragg peaks out of the energy region of interest. The availability of nanocrystalline diamond anvils has reduced this problem for experiments on both energy-dispersive and energy-scanning XAFS beamlines (for crystalline germanium at high pressure, for example, see Baldini *et al.*, 2011).

Many accurate XAFS experiments under high-pressure and high-temperature conditions have also been carried out using large-volume cells. After the initial application of the Paris– Edinburgh (PE) press to liquid and solid HgTe (Katayama *et al.*, 1997), many studies of metals, glasses, molecular fluids and solutions have been carried out using similar methodologies, as shown for example in an accurate study of solid and liquid tin (Di Cicco *et al.*, 2006). Further details and other applications of XAFS using large-volume cells are reported in Shen & Wang (2014).

As an example of the application of XAFS to matter under variable pressure and temperature conditions, we mention here some results related to simple polymorphic elemental substances such as germanium and gallium. In Fig. 3 the phase diagrams of the two substances are reported. The coexistence lines related to the stable thermodynamic phases in the selected (P, T) regions are those reported previously (see Tonkov & Ponyatovsky, 2005 and references therein) although some uncertainty still exists. Both systems are ice-type elements with a large degree of polymorphism; we refer to the

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original publication (Tonkov & Ponyatovsky, 2005) for details. In Fig. 3 we have also included shaded regions in which metastable phases such as undercooled melts, amorphous states and other crystalline structures are known to exist (for gallium, see Di Cicco, 1998; Comez *et al.*, 2002; for germanium, see Di Cicco & Filipponi, 2015; Di Cicco *et al.*, 2003). In this short chapter, we include Ge and Ga *K*-edge XAFS data collected under the selected thermodynamic conditions indicated by the symbols in Fig. 3.

In Fig. 4, we report the raw near *K*-edge X-ray absorption data of crystalline (c-Ge), amorphous (a-Ge) and liquid (l-Ge) germanium. Data of high quality were obtained using a large-volume PE press, and the sensitivity of XAFS to the local



Figure 3

Phase diagrams of germanium (left) and gallium (right) at high pressures and temperatures. The coexistence lines (in red) are taken from Tonkov & Ponyatovsky (2005) (slightly different results have been found by several authors). Dotted (red) regions indicate that undercooled liquid germanium and gallium can be obtained as metastable phases on cooling the melt. Dashed (black) regions indicate that germanium and gallium can be found in other metastable solid phases (amorphous or crystalline). The pressures and temperatures corresponding to the XAFS experiments reported here are indicated by symbols (\times , +) in different colours.



Figure 4

K-edge region of the XAFS spectrum of crystalline (c-Ge), amorphous (a-Ge) and liquid (l-Ge) germanium at different pressures and temperatures (see the points in the phase diagram in Fig. 3).

structure of the different aggregation states can clearly be appreciated. The discontinuous red shift and the spectral changes occurring upon melting reflect the metallic character of the liquid state. The weaker XAFS features observed in a-Ge compared with c-Ge reflect the loss of long-range ordering. Further details can be found in Di Cicco & Filipponi (2015) and references therein.

The results of a high-pressure XAFS experiment on liquid and solid gallium, using a heatable DAC and an energydispersive XAFS setup, are reported in Fig. 5 (for further details, see Comez et al., 2002). Two isothermal pressurizations were carried out on gallium samples, and the XAFS results reflect the modification of the local structure in the different liquid and solid phases. The experiment also included X-ray diffraction, and the transitions were also easily monitored by changes in the near-edge structures (Comez et al., 2002), similarly to the case of germanium. Gallium is characterized by the ease of formation of metastable phases, with exceptional undercooling capabilities and the presence of multiple crystalline phases in different portions of the phase diagram. For example, Ga III can be found at room temperature in the stability region of Ga II (Ga III is also the stable phase above 20 GPa; Schulte & Holzapfel, 1997) and X-ray diffraction is often unable to detect specific peaks for the various phases.

In this context, XAFS can be extremely useful to detect phase transitions, providing determination of the local





Left: Ga K-edge XAFS experiments on liquid and solid gallium at variable pressures and temperatures (experiments and best-fit calculations are shown in blue and green, respectively). Further details can be found in Comez *et al.* (2002). XAFS spectra at room temperature (298 K) are reported in the top left panel; the crystallization of gallium at 2.5 GPa can easily be observed (the dashed line is a guide for the eye). In the lower left panel we report the XAFS spectra at 378 K, where crystallization is observed above 4.7 GPa. The effect of the increased temperature is evident both in the XAFS intensity and in the transition pressure. Right-hand panel: reconstruction of the pair distribution functions g(r) in liquid and solid gallium based on XAFS data at 298 K (upper right panel) and 378 K (lower right panel). Differences in the short-range distributions are clearly detected at increasing pressures and temperatures, confirming the sensitivity of the XAFS technique.

structure. In Fig. 5 crystallization of the high-pressure solid phases can easily be appreciated by a drastic change of the main XAFS signal frequency related to the average firstneighbour interatomic distance (see the dashed line in Fig. 5 as a guide for the eye). The experimental XAFS spectra (in blue) were of good quality, and the noise and distortions (Bragg reflections) typical of energy-dispersive configurations using DACs were kept to a minimal level. The XAFS spectra were analyzed (Comez et al., 2002) by modern data-analysis techniques (best-fit curves are reported in green in Fig. 5), providing structural parameters that were useful for reconstruction of the short-range pair distribution function in solid and liquid gallium. Structural results are reported in Fig. 5 (left panels), showing the peculiar sensitivity of XAFS to changes in the local structure. The effect of both pressure and temperature as thermodynamic parameters is clearly seen, with distinct shifts of the average interatomic position and broadening of the main first-neighbour peak.

The mentioned applications of XAFS to germanium and gallium under variable pressure and temperature conditions clearly show the sensitivity of this technique to transitions in condensed matter and its important potential for determination of the local structure, which can be particularly useful for studying metastable and transient states.

Much effort is currently being devoted to expanding the (P, T) range for XAFS experiments. Micro-sized beams and time-resolved and high-pressure techniques may help in increasing the attainable high pressures in static and time-resolved DAC experiments by a factor of ten.

Maximum pressures well above 100 GPa have now become accessible (in the 500 GPa range with a two-stage DAC; Dubrovinskaia *et al.*, 2016; Sakai *et al.*, 2018) and XAFS experiments in this pressure range have become possible. Laser heating, dynamic shockwaves and the use of fourthgeneration sources also offer the possibility of performing (transient) XAFS measurements of matter in region (8) of Fig. 1 and beyond in the so-called warm-dense-matter regime. For example, applications to iron melting at high pressure and temperature using synchrotron (Aquilanti *et al.*, 2015) or freeelectron laser (Harmand *et al.*, 2015) sources indicate that XAFS applications are being gradually extended to pressure and temperature ranges that are presently extremely challenging or just unreachable with current technologies.

Disclaimer. Owing to the extensive field covered by this chapter, we were forced to select references for inclusion in the bibliography. Regrettably, there will be important papers that have not been cited and we apologize to the colleagues involved.

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