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Optic modes in the AlPdMn icosahedral phase

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Abstract. The dynamics of the icosahedral phase AlPdMn have been reinvestigated on a centimetre-size single grain using inelastic neutron scattering measurements. As previously found, well defined acoustic modes are observed close to strong Bragg reflections. Transverse acoustic phonons remain resolution limited for wavevectors smaller than 0.35 $Å^{-1}$ and a broadening occurs for larger wavevectors. Beside acoustic modes, the scattering function $S(Q, \omega)$ shows an extremely rich structure. A series of broad (1 THz) dispersionless excitations appears with energies close to 1.8, 3, 4 and 5.5 THz. These excitations were found at different points in reciprocal space and present intensity variation. Some of these 'optic' bands might be associated with crossings at quasi-Brillouin zone boundaries. The system has also been investigated at high temperatures up to 800 °C. Beyond a 10% reduction of the sound velocity, results are identical to those obtained at room temperature. In particular the transverse acoustic modes remain resolution limited for wavevectors smaller than 0.35 $Å^{-1}$.

1. Introduction

The dynamical properties of quasicrystals have been studied theoretically and experimentally (see [1–3] for an introduction to the subject). Of particular interest are the 'perfect' quasicrystals of the AlCuFe and AlPdMn systems [4,5]. These quasicrystals exhibit extremely sharp Bragg reflections corresponding to correlation lengths of several tenths of a micrometre [10], similar to what is obtained for periodic crystals [6,7]. Moreover, centimetre-size grains can be grown in the AlPdMn system via classical methods [7–9]. The quality of these large grains is such that dynamical diffraction is observed on a macroscopic scale. In particular, the Borrmann effect, normally associated with crystals of high perfection, has been experimentally shown to occur on a single grain of AlPdMn [10].

The atomic structure of quasicrystals is now best understood via higher-dimensional crystallography [1–3]. Following this approach, atomic models have been proposed for AlCuFe [11] and AlPdMn icosahedral phases [12–14]. The results present a 'low-resolution' image in that the details of the atomic structure are not yet specified. However, clear insights are obtained about the existence of well specified atomic clusters, which are packed quasiperiodically into hierarchical aggregates [15].

Such realistic models allowed the calculation of dynamical properties that should be close to what is encountered experimentally. Calculations have been proposed by Hafner

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and Krajci [16–18] on the basis of relaxed atomic models with realistic pair potentials for AlZnMg and AlLiCu periodic approximants of the icosahedral phase. The main conclusions are similar to the early calculations by Los *et al* [19, 20] of the dispersion relations for several periodic approximants of the 3D Amman tiling. There are well defined acoustic modes close to the strong Bragg reflections which define Brillouin zone centres. Quasi-Brillouin zone boundaries are defined around high-symmetry points in the reciprocal space [21, 22], and are packed hierarchically around the zone centres. Acoustic branches experience gap openings when crossing these quasi-Brillouin zone boundaries, with the widths of the gaps increasing with increasing wavevector. These calculations also produce numerous optic branches with a density that is especially large in the higher-frequency range.

Similar results were obtained by Poussigue *et al* [23] for the calculation of the dynamical response of an AlMnSi phase, an alloy similar to the i-AlPdMn phase. They found sharp acoustic modes close to the Bragg reflection, the width of which increases as was observed experimentally in the AlPdMn phase.

Experimentally three icosahedral phases, of different atomic structures, were previously investigated: i-AlLiCu [24, 25], i-AlCuFe [26, 27] and i-AlPdMn [28]. The isotropy of the acoustic modes has been confirmed in these systems, and was also observed in the cubic approximant phase R-AlLiCu. Dispersion relations were measured showing quasi-zone boundaries in the i-AlLiCu and i-AlPdMn phase. The higher-energy modes in the icosahedral AlLiCu and its cubic approximant R-AlLiCu phase showed differences which are consistent with an enhanced degree of localization of these excitations in the icosahedral phase [25]. Acoustic phonons measured in the i-AlPdMn phase showed a significant broadening for wavevectors larger than 0.35 $Å^{-1}$ which was interpreted as the consequence of a continuous distribution of dispersionless 'optic' modes in the range 2–4.5 THz [28].

In this paper we present new measurements carried out on a single grain of the i-AlPdMn phase. A 3 cm^3 single grain was obtained by the Czochralski method [9]. It is ten times larger than the sample previously used [28], and presents a very narrow mosaic distribution (0.06°). This allows new insights into the dynamics of the icosahedral phase, particularly in the high-resolution study of acoustic modes and in the study of optic modes (some of these results were presented in [53]).

The paper is organized as follows. Details of the inelastic neutron scattering experiments are presented in section 2. Results of measurements in the acoustic regime are presented in section 3, and results on the 'optic' modes are presented in section 4. In section 5 we discuss the results of a temperature study of the dynamical properties. Even when close to the melting point (800 °C), the dynamical properties are identical to those obtained at lower temperature apart from a 10% decrease of the sound velocity.

2. Experimental details

The sample used in the present investigation is a single grain obtained by the Czochralski method, in the form of a cylinder 1 cm in diameter and 2.5 cm long. Its single-grain character and homogeneity were carefully checked by γ-ray and neutron scattering techniques, revealing a mosaic width of 0.06° in the scattering plane and 0.03° out of the plane [9].

Neutron inelastic scattering measurements were performed on a triple-axis spectrometer. In a first step, excitations were measured on the triple-axis spectrometer 1T at the Orphée reactor (Laboratoire Léon Brillouin, France) using a pyrolytic graphite monochromator and a bent analyser leading to an energy resolution of about 0.35 THz. Relaxed resolution was accepted for measuring high-energy excitations which correspond to rather weak signals. Constant-k_F (2.662 $Å^{-1}$) scans were carried out at room temperature and 600 K to enhance the weak signal of the high-energy excitations [27, 28]. Measurements at 600 K did not provide any evidence for changes in the positions and widths of the excitations. Measurements were also carried out at higher temperatures, up to 1070 K.

Low-energy acoustic phonons were measured on the triple-axis 4F2, using the cold source of the reactor. Because of the better energy resolution achievable when working at a constant k_F equal to 1.64 Å^{-1}, acoustic phonons were measured down to wavevectors of 0.05 Å^{-1} . The instrumental energy resolution was about 0.12 THz, three times better than what was achieved on 1T. (One should note, however, that the resolution is still relatively low. This is because the acoustic slope does not match the resolution ellipsoid, which precludes the full benefit of the focusing effect in the transverse geometry.) These last measurements were carried out at room temperature.

3. Data analysis in the acoustic regime

In the long-wavelength limit ($|q| \rightarrow 0$) the continuum approximation applies to quasicrystals as to any other condensed matter system. Well defined, propagating, collective excitations are expected to be observed in the form of phonon branches. It is also predicted that the transverse acoustic phonons reduce to a single degenerate branch, due to icosahedral symmetry. In the acoustic regime, the dispersion relation is linear and given by

$$\omega_{T(L)}(q) = v_{T(L)}|q| \tag{1}$$

where T and L stand for transverse and longitudinal modes respectively.

A Taylor expansion of the neutron scattering differential cross-section gives the response function:

$$S_{T(L)}(Q,\omega) = \frac{\langle n(\omega) + \frac{1}{2} \pm \frac{1}{2}\rangle}{\omega_{T(L)}(q)}(e_{T(L)} \cdot Q)^2 F_{el}^2(Q)\delta(\omega \pm \omega_{T(L)}(q)) \tag{2}$$

where Q is the momentum transfer, G is a vector of the reciprocal lattice (zone centre) and the phonon wavevector is given by $q = Q - G$. $e_{T(L)}$ is a polarization vector orthogonal (parallel) to the phonon wavevector q for transverse (longitudinal) modes and $F_{el}(Q)$ is the elastic structure factor of the quasicrystalline structure. Figure 1 shows relatively strong Bragg peaks around which phonon measurements were carried out. These points were chosen as Γ points (or zone centres) around which extended dispersion relations were constructed (for a complete discussion of these definitions see [25] and [28]).

Figure 2 shows the dispersion relation for wavevectors in the transverse geometry around the Bragg peak D lying on a twofold axis (labelled 52/84 in the Cahn *et al* scheme [29]). For q smaller than 0.35 Å^{-1}, well defined excitations are measured with a linear dispersion relation according to (1) and (2). The energy width of these excitations is limited by the instrumental resolution (i.e. 0.35 THz). This is the only region where there is a one to one correspondence between the measured scattering function $S(Q,\omega)$ and a phonon branch. Measurements in the low-q regime (0.05 to 0.35 Å^{-1}) have been performed on the 4F2 triple axis around the Bragg peak A. Similar to what was obtained on 1T, the acoustic phonons have a resolution-limited width of the order of 0.12 THz. These additional points have been reported also in figure 2. This is justified by the isotropy of acoustic modes in icosahedral phases. The slope of the linear part of these dispersion curves is in good agreement with the sound velocity as deduced from ultrasonic measurements [30].

The main quasi-Brillouin zone boundaries are shown in figure 2 [28]. When the acoustic branch crosses such a pseudo-zone boundary one expects a gap to open. The first important quasi-Brillouin zone is located at 0.35 Å^{-1}. At this wavevector the dispersion relation

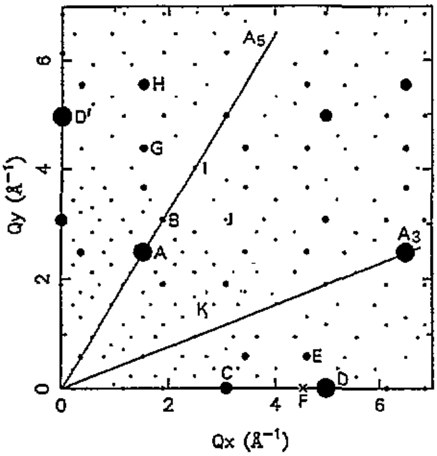

Figure 1. Twofold scattering plane of the AlPdMn icosahedral phase. Sizes of spots indicate intensity of the reflections.

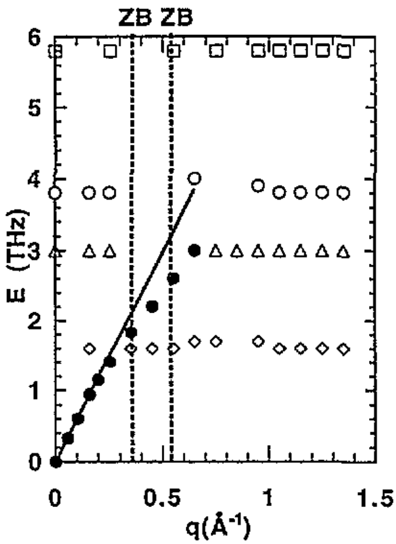

Figure 2. Dispersion relations for excitations measured in the transverse geometry from point D. Black dots correspond to acoustic modes (see text). The solid straight line corresponds to the linear acoustic dispersion as deduced from ultrasonic sound velocity measurements. The different symbols correspond to the different maxima of the 'optic modes' identified in the measured signal. The main pseudo-Brillouin zone boundaries, as determined from the 6D description of the quasicrystal, are indicated with the symbol ZB.

departs slightly from the linear regime, but no gap could be found even with the high-resolution measurements carried out on the 4F2. This implies that a gap, if any, must have a width smaller than 0.04 THz.

For values of q larger than 0.35 Å^{-1} the signal broadens and the dispersion relation is no longer linear. A fit of the experimental response functions with a damped harmonic oscillator lineshape leads to a determination of position and width of these excitations. From this point other excitations start to mix up with the acoustic signal. Indeed, using relation (2) it is possible to compute from the fit a normalized intensity, which depends only on F_{el}, and no longer on q. In the acoustic regime this normalized intensity is constant, and this is what is observed for q smaller than 0.35 Å^{-1}. Above this wavevector, if a single excitation is used to reproduce the data the normalized intensity shows a significant increase, indicating that one is mixing several phonon contributions. However, up to a q of about 0.65 Å^{-1} it is possible to disentangle the acoustic contribution from the other broad excitations. This 'acoustic' signal is reported as a black dot in figure 2. (Such separation of acoustic and optic modes could not be carried out with the previous experiment [28] because of the weak optic mode signal: only a single contribution was reported in [28], which explains the differences in the dispersion curves.)

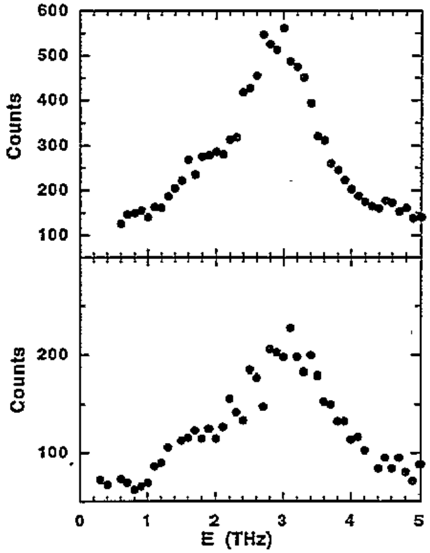

Figure 3. Constant-Q energy scan taken at q = 0.65 Å^{-1} from the Bragg peak D in transverse geometry (top) and at point K, a place in reciprocal space away from any strong Bragg peak (bottom). Two excitations at 1.8 and 3 THz are visible.

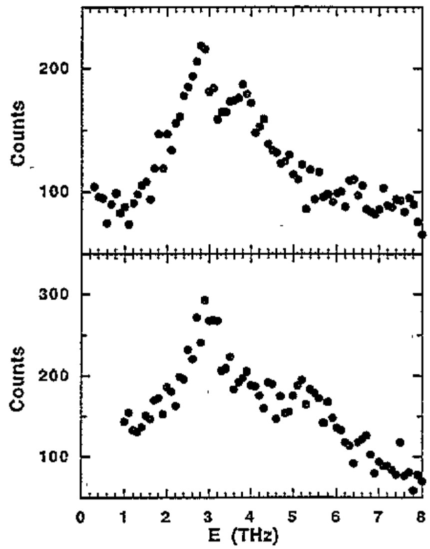

Figure 4. Constant-Q energy scan taken at the Bragg peak I, on a fivefold axis (bottom), and at the Bragg peak J (top). Although the two points are relatively close in reciprocal space the excitation located at 5 THz shows significant relative intensity variation.

4. Evidence for optic modes

Taking advantage of the large size of the sample, very weak signals have been measured with acceptable accuracy. In particular, four broad excitations (~1 THz, i.e. three times as large as the instrument resolution) were observed at energies of about 1.8, 3, 4 and 5.5 THz. An example of the corresponding signal is shown in figures 3 and 4. Whereas the position of the excitations at 1.8, 3 and 4 THz is relatively well defined, the excitation at 5.5 THz is very broad, and difficult to locate precisely. The two main experimental results concerning these excitations are the following.

(i) They can be found almost anywhere in the reciprocal space. This is illustrated in figure 3, where the excitations located at 1.8 THz and 3 THz are measured, as well close to point D and point K, although these points are far away in reciprocal space. Note that point K, with coordinates (2.56, 1.42) Å^{-1}, has been chosen to be away from any strong Bragg peak, demonstrating that this signal is not related to the presence of a strong zone centre.

(ii) These excitations present significant intensity variation when moving in reciprocal space, as expected from coherent inelastic scattering. This is illustrated in figure 4 which presents constant-Q energy scans measured at points J and I. These points are relatively close in reciprocal space, but the excitation centred at 5.5 THz has almost vanished when going from J to I. (Note that a measurement carried out on a powder sample would have mixed up these two different contributions.)

These excitations are characterized by flat branches as shown in figures 2 and 5. As discussed in [28] they cannot be attached unambiguously to any Bragg reflection and deductions from (2) (or a similar equation) are no longer valid. This precludes the definition of the wavevector and makes unphysical any description of the modes in terms of a propagating wave. Moreover these broad excitations might result from a distribution of phonons having close energies. The flat broad modes are referred to as optic modes hereafter.

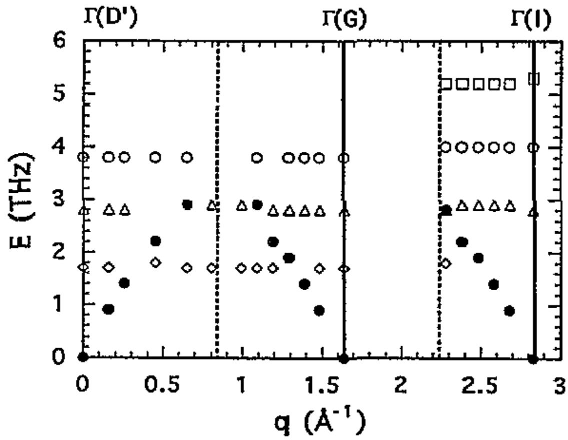

Figure 5. Dispersion relation for excitations along the line D'GI. The different symbols correspond to the different maxima identified in the measured signal. The full dots are for the acoustic signal.

Nevertheless, it may be of interest to present the results globally in the form of a pseudoextended dispersion relation, with selected strong Bragg reflections taken as quasi-zone centres (Γ points) [21, 22, 28]. This is shown in figures 2 and 5, along with data from ultrasonic measurements. As already pointed out in section 3, no evidence of a gap at the crossing of the pseudo-Brillouin zone boundaries has been given. However, optics modes have energies that almost correspond to those of the crossing points of the acoustic branch with quasi-Brillouin zone boundaries. This is observed for the two optic modes at 1.8 and 3 THz which, then, may result indirectly from gap opening.

As already mentioned, well defined acoustic modes and standing modes showing up as flat branches at crossings with quasi-Brillouin zone boundaries have been predicted by Krajci and Hafner [18], though for an AlLiCu model whose structure departs significantly from that of the AlPdMn phase. Also in the calculations by Poussigue *et al* [23] the response function $S(Q, \omega)$ broadens at quasi-Brillouin zone borders which is interpreted in terms of criticality of the modes. Moreover, the weak excitation at 1.8 THz is indeed given by the calculations. The conclusions of these calculations are significantly different for low-order approximants (1/1 and 2/1) which suggests that the above features are typical of quasiperiodicity.

Finally the present results are also in agreement with the general vibrational density of states (GVDOS) measured by Suck on a powder sample [31]. The GVDOS shows a main band at about 4 THz which may be split in two subbands at about 3 and 4 THz. A weak third band is observed at 5.3 THz with a shallow pseudogap at 5 THz. The scattering function $S(\boldsymbol{Q}, \omega)$ extracted from these powder measurements also shows dispersionless optic modes around 2 THz as observed with the present data.

5. Temperature dependence of the dynamical properties

The same measurements were carried out up to 800 °C on the 1T triple axis, i.e. 60 °C below the melting point. Investigations of the dynamical properties at high temperature are interesting for several reasons: phonon–phason coupling, diffuse scattering and mechanical properties.

Phason modes correspond to long-wavelength fluctuations of the cut in the 6D description of quasicrystals [32–40]. The elasticity theory of quasicrystals predicts an anisotropic broadening of acoustic phonons if a phonon–phason coupling occurs [37–39]. Experimentally, Coddens *et al* found evidence for an atomic jump above 650 °C in the AlCuFe phase, interpreted as 'phason hopping' [46, 47]. At high temperature it is believed that phason modes become effective, the system being in a random tiling phase (unlocked phase) [46, 47]. If this is the case one should see an anisotropic broadening of acoustic phonons when the temperature is increased. Moreover it has been shown that at room temperature there are frozen-in long-wavelength phason fluctuations leading to diffuse scattering located close to Bragg reflections, in agreement with the elasticity theory [48–50]. Preliminary results showed that this diffuse scattering partly disappears at high temperature [51]. This is also a good reason for expecting a phonon–phason coupling.

Finally, anomalous mechanical properties close to the melting point have been observed. Whereas the i-AlPdMn phase is brittle up to 700 °C, the phase shows a large plasticity above 750 °C [8, 52]. The mechanism of this plastic deformation is not yet understood and it is certainly interesting to collect information on the elastic properties of the icosahedral phase at high temperature.

The sample was slowly heated up to 800 °C while Bragg peaks and diffuse scattering were measured. Some changes in the Bragg peak intensity and diffuse scattering were observed and will be reported in a forthcoming paper [51]. The main results of interest for the present dynamics study are that the AlPdMn phase remains icosahedral, and no new phases at elevated temperatures were observed.

For dynamical properties, results are very simple to summarize since they are identical to what is obtained at lower temperature! At 800 °C the slopes of the acoustic branches are slightly smaller (about 10% smaller) for transverse modes leading to a transverse sound velocity equal to 3390 m s^{-1} as compared to 3700 m s^{-1} at room temperature. This means that elastic constants do not change significantly. At the resolution achieved with 1T, the transverse acoustic phonons remain resolution limited for wavevectors smaller than 0.35 $Å^{-1}$. A broadening, similar to what is observed at low temperature, occurs for larger wavevectors. One should note, however, that the energy window of this experiment is large, which means one is looking at relatively fast process compared to atomic self-diffusion, for instance. The resolution of 0.35 THz means that it is impossible to look at broadening smaller than 0.1 THz. This corresponds to a characteristic time t equal to 10^{-11} s, or to a characteristic propagation length (when using the transverse sound velocity) equal to 360 Å. This might not be sufficient to 'see' the long-wavelength phason fluctuations, if they are present.

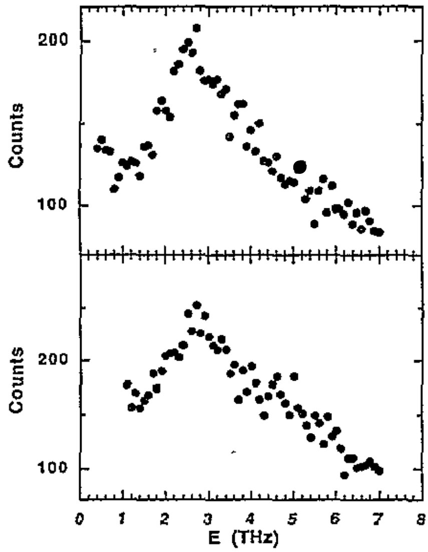

Figure 6. Same as figure 4 for a temperature equal to 800 °C. The data have been corrected for a Bose occupation factor for a direct comparison. There are no changes in the spectrum.

Optic modes also did not show any changes when compared to the results presented in section 4. The relative intensity of the four optic modes was found to be identical, even for the 1.8 THz mode. This is illustrated in figure 6, where the constant-Q scans at points J and I have been represented. To allow for a direct comparison with figure 4, data have been corrected for a Bose occupation factor.

6. Conclusion

The dynamical properties of the icosahedral AlPdMn phase have been studied on large single grains by means of triple-axis measurements. Close to strong Bragg reflections there are well defined acoustic modes. The width of the measured signal broadens rapidly for wavevectors larger than 0.35 $Å^{-1}$. Four dispersionless excitations have been found. These are broad excitations (1 THz) located at 1.8, 3, 4 and 5.5 THz. The excitations located at 1.8 and 3 THz may be associated with the crossing of pseudo-Brillouin zone boundaries.

Studies at 800 °C did not show any changes, apart from a 10% reduction in the transverse acoustic slope. The acoustic phonons remain resolution limited below 0.35 $Å^{-1}$ and the optic modes show the same reciprocal space and intensity dependence.

It is not possible to conclude definitively from the present experimental results whether the 'optic modes' are localized or critical. This should emerge from comparison with calculations on a detailed model of the structure. The large number of data available will certainly strongly constrain any model and, hence, generate progress in the understanding of the influence of quasiperiodicity on dynamics.

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