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Names and affiliations of applicants (*indicates experimentalists):

Javier Perez* Dominique Durand,* Jean Doucet
LURE, Bat 209D, Universite Paris-Sud, F-9 1405 Orsay Cedex

Claudio MasciovecchioyFrancesco Sette
ESRF, Avenue des Martyrs, F-38043 Grenoble Cedex

Report:

The first part of our beam-time (4 days) was devoted to the measurement of an acoustic branch of TBBA, an organic material for which the velocity of sound is expected to be close to that of a protein crystal. The idea was to check whether ID16 resolution was enough to measure sound velocities lower than 2000 $m.s^{-1}$ in organic materials. In the second part (2.5 days), we made preliminary measurements on a lysozyme monoclinic crystal.

Experimental conditions

All measurements were carried out at room temperature with λ=0.57 A, corresponding to the Si(11,11,11) reflexion in back-reflexion geometry. The overall instrumental energy resolution was 1.5 meV (FWHM), well approximated by a lorentzian shape.

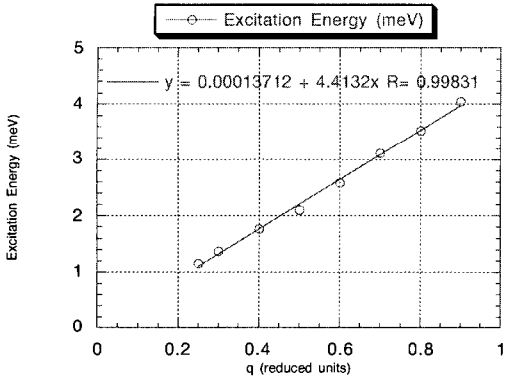
The TBBA samples (A2/a, a=17.57Å, b=5.75Å, c=53.2Å, β=115.47°) were plate-shaped (≈ 300 micrometers thick) with typical dimensions of 8 x 8 mm^2.

The lysozyme crystal ($P2_1$,a=28.0Å, b=62.5Å, c=60.9Å, β=90.8°) of dimensions 4 x 2 x 0.5 mm^3, was inserted into a closed steel cell with controlled humidity, and pre-oriented at the Institut de Biologie Structurale.

Results for TTBA

Two collections along the c* direction (at (0 0 2.65) and (0 0 3) nodes in reduced units of reciprocal space) showed highly damped excitations respectively at about 2.8 and 3.75 eV. These excitations were difficult to fit, due to an important central peak. We decided to perform the next measurements along the a* direction.

Energy scans were collected along a* at h=4.25, 4.3, 4.4, 4.5, 4.6, 4.7, 4.75, 4.8 and 4.9, in reduced units. The data were fitted with a narrow Lorentzian centered at $\omega=0$ to account for elastic and quasi-elastic scattering and a double Lorentzian (including Bose-factor) centered respectively at ω_0 and $-\omega_0$, accounting for the phonon excitation.



The figure represents the value of ω_0 as a function of q, where q=h-4 in reduced units. A linear fit correctly accounts for the data, driving to a slope of 1.11 meV.nm (with a*=3.96 nm^{-1}). The velocity of propagation is directly proportionnal to the slope : v ($m.s^{-1}$) = 1520 * slope (meV.nm). The value found for v is : 1690 $m.s^{-1}$.

This value is lower than that determined by coherent neutron scattering (Doucet thesis, 1978), 2500 $m.s^{-1}$. The reason, most probably, lies in the fact that in our fit, we integrate a true acoustic phonon together with an optical one. This is clearly visible in the spectra with high values of q (not shown). For example at q=0.8, a fit with two double Lorentzian leads to values of ω_1 and ω_2 of 2.46 meV and 3.94 meV, respectively, instead of 3.52 meV with only one double Lorentzian.

The normalized excitation intensity was fitted to a decreasing power function as a function of q, leading to a power value of, - 1.8, while in theory, an acoustic excitation should drive to a value of, - 2. The lower value found here is presumably also due to the "parasitic" optical component.

It would certainly be possible to separate the two contributions for most values of q, provided that data are collected with longer exposition.

Preliminary results for lysozyme

Experiments with lysozyme were made difficult essentially because of technical problems from the ESRF ring : one day of beam was lost because of a vacuum incident in a ring cavity, which, moreover, forced us to work afterwards with a very short life-time beam.

Energy scans were nevertheless performed along the a* axis, between the (2 0 0) and (3 0 0) nodes. Although difficult to characterize quantitatively because of an important central peak, phonon excitations are present. These excitations should be correctely measurable with enhanced experimental conditions :

l/ A larger crystal : Much time was spent in setting up the growth conditions of very big crystals. The crystal we used had grown for only one month. It is certainly possible now to get larger crystals.

2/ More statistics : Due to the decision of measuring first on the TBBA sample, less time was available for the lysozyme crystal. With the same amount of beam-time dedicated exclusively to lysozyme, we could certainly get a much better statistics for low intensity excitations.

3/ Very narrow angular steps : Performing measurements with very narrow steps in q, in order to follow very precisely the evolution of the excitation, would probably be the best way to be sure that the excitation is correctly described as an acoustic mode.