Phonons are quanta of elementary vibrational motions in solids and their energy and momenta distributions directly determine many fundamental thermodynamic properties of solids, such as heat capacitance or heat conductivity. Closely linked to the local arrangement of atoms, interesting new properties arise when symmetries are broken and surfaces are created, making surface phonons becoming strongly dependent on the object geometry [9, 16].

Surface phonons have recently attracted much attention because of their counter-intuitive physical properties and their promising applications in photonics and nanophotonics from the mid-IR (3-8 µm, 155-413 meV) up to the far-IR (15-1000 µm, 1.2-83 meV) [10]. For example, materials such as SiC possess strong surface phonon resonances and may exhibit a highly coherent emission upon heating. This behaviour differs completely from the conventional incoherent black-body radiation [17]. At the nanoscale, two surfaces sensing each other’s near-field, may exhibit strongly enhanced heat transfer due to the presence of surface phonons [18]. These strongly modified emission and absorption properties may be favorably applied to the design of phononic metamaterials acting as extremely efficient passive coolers [19] or to the generation of improved detectors and emitters in the mid-IR, especially when surface-phonons interact with plasmons like in graphene-type materials [20].

Beyond that, surface phonons concentrate electromagnetic energy at deep sub-wavelength scales in the same way as surface plasmons, but up to the far-IR region, resulting in further intriguing nanophotonic applications and techniques [21]. SEIRA (surface enhanced infra-red absorption [22]) for instance, harnesses the strong and local enhancement of the infra-red electromagnetic field to probe molecular vibrations [11]. This enhancement, usually ascribed to surface plasmons, can be significantly magnified by the exceptionally high-quality factors of surface phonons [11], leading to potential new approaches in biology [12]. Finally, metamaterials made up of surface phononic materials are key for super-resolution lenses in the mid-IR [23]. Strikingly, all these applications rely on the nanostructured electro-magnetic field in the vicinity of surfaces of metamaterials or nanoparticles. Designing or even engineering the electro-magnetic local density of states (EMLDOS) for specific functionalities require however the unambiguous visualization of such field modulations at the nanometer scale. This became accessible by near-field infra-red techniques [1-4, and more recently by electron energy loss spectroscopy (EELS) in a scanning transmission electron microscope (STEM) [5, 6]. EELS can measure vibrations at the atom scale [3, 7, 8] and reveal dispersion relations of phonons [24, 25]. Nevertheless, intrinsic to those techniques, they only allow for two-dimensional imaging and do not provide directional field information from the start. Recently, in the visible range, tomographic tilting of plasmonic EELS data in combination with sophisticated eigenmode-based reconstruction algorithms have subsequently led to a series of publications [26–29], culminating in the full, three dimensional and vectorial reconstruction of plasmonic field at all frequencies in the visible regime [30, 31]. The possibility to perform such a reconstruction is deeply rooted in the genuine relation between plasmon excitations and their EMLDOS [32], which describes the variation of the square modulus of the eigenfields, projected along arbitrary axes, in space and energy.

Given the strong formal analogy between surface plas-
Figure 1. **Tomographic surface phonon EELS experiments.** a. Surface phonon tomography set-up. MC: Monochromator. b. High angle annular dark-field images of an MgO cube acquired at two different tilt angles. c. Selected spectra for the two different tilt angles taken at the positions indicated on b. Note the difference of spectra upon tilt for a fixed beam position (modes II and III).

We describe the experimental set-up in figure 1a. A 60 keV electron beam with an initial energy width of $\approx 350$ meV is filtered by a monochromator to obtain a final energy spread around 7 to 10 meV. This monochromator efficiently optimizes the current left after monochromation, with a beam current of a few pA in a sample area of $\approx 1 \text{ nm}^2$. The nano-object presented in figure 1 is a MgO cube with edge length of 191 nm deposited on a 20 nm thin Si$_3$N$_4$ substrate. The back surface of the substrate was covered with a few nm thin carbon layer to avoid charge-related issues (see Methods). By scanning the electron beam, one can collect high angle annular dark field (HAADF) images that reveal the morphology of the cube. Sample tilting by an angle $\alpha$ allows imaging of the cube under different orientations (Figure 1b). At each position of the scan, an EELS spectrum was recorded. Figure 1c shows clear spectral responses in the upper far-IR, extracted at two different tilt angles for two different positions of the electron beam. As the spectral features do change with both the electron beam position and the tilt angle, the selected spectral, spatial and tilt resolutions of the used setup (see Methods) has proven adequate to resolve directly signals characteristic of the main modes of cubes [6, 9]. In order to understand their physical origin of the three main spectral features, numbered I, II and III, we systematically recorded EELS spectral-images (SI) at different tilt angles. For a first analysis, we present in figure 2a energy maps filtered at the energy maxima of the modes I, II and III, for two different tilt angles (see full tilt maps in the supplementary information). These maps are generated by using a fitting routine for each spectral mode of a SI (see Methods), and writing the resulting intensity in the filtered image pixel.

The principal spatio-spectral features are already apparent. The MgO cube exhibits signals strongly localized in energy at 68 (mode I), 69 (mode II) and 78 meV (mode III). When the electron beam is propagating perpendicular to the faces that are parallel to the substrate (0 mrd tilt), mode I is localized on the four corners. Tilted spectral-imaging directly shows a difference in intensity between the signal on corners in vacuum and those on the substrate. The latter is much weaker than the former. Mode II is not directly seen on the 0 mrd map, but becomes visible upon tilting (see also supplementary material). In this case, the signal originates from the edges, and again the signal is much weaker for the edges attached to the substrate. Finally, the mode III is present at all angles. However, its spatial distribution is more difficult to understand.

With the exception of mode I, which can be assigned to the corner, an unambiguous interpretation of the data is not straightforward. In fact, the signal of mode II is essentially revealed for geometries where the beam direction is not parallel to an edge. Such an angular dependence points to the vectorial nature of the signal. Furthermore, the localization of the signal of mode III cannot be deduced directly from the maps. As presented in Figure 2b and in the supplementary material, we performed simulations for a cube of equal size in vacuum with the boundary element method (BEM) [33] using the MNPBEM package [34]. The simulations reveal similar features as the experimental data and confirm the existence of three different modes. The main deviations are...
the absence of signal inside the cube in the experimental data and the absence of asymmetry in the theoretical data. The former relates to the experimental issue of electrons getting scattered out of the spectrometer due to the large thickness. The latter is due to the fact that the substrate influences have not been accounted for in the simulations.

In order to understand the full surface phonon response of the cube, we used a tomographic reconstruction scheme. Owing to the mathematical similarity between the surface plasmons and phonons, we adapted the methods recently developed to measure the plasmonic EMLDOS [35] (see Methods and supplementary material) to the quasistatic limit. The quasistatic limit applies because the typical free space wavelengths of the surface phonons (several tens of μm) are much larger than the cube size. This leads to a simple description of the vibrational response in terms of well-defined eigenmodes with the following underlying principle. The vibrational response of the cube can be described as a set of modes indexed by a number \( k \), \( \lambda_k, u_k(s) \), where \( \lambda_k \) is an eigenvalue, and \( u_k(s) \) the spatial distribution of the associated eigencharge (\( s \) being the surface coordinate) [6, 9]. From this set, all linear physical quantities can be deduced in this quasistatic limit [9, 36, 37]. This includes EELS and the EMLDOS, which are both linearly related to (see supplementary material):

\[
A(s, s') = \sum_k \left( \frac{\lambda_k + \frac{1}{2}}{\Lambda(\omega) + \lambda_k} \right) u_k(s) u_k(s')
\]  

(1)

where \( \Lambda(\omega) \) is a material-dependent function that directly depends on the dielectric properties of the material at frequency \( \omega \). The \( \omega \) dependent prefactor determines a dispersion relation for the eigenvalues \( \lambda_k \) so that a distinct spectral signature dominated by the surface phonon energy can be attributed to each mode [9, 38].

Due to this relation, a robust procedure for the reconstruction can be envisaged. First, we measure EELS SIs for different tilt angles. Then \( \lambda_k, u_k(s) \) can be retrieved from the EELS data. This is done by fitting the EELS signal to its theoretical expression (see supplementary material) which is expressed as a function of equation (1). To speed up the reconstruction, the sought eigenvectors \( u_k(s) \) are projected on the eigenvector basis of an ideal structure \( \tilde{u}_k(s) \). In the case of a cube, the ideal structure is a cube in vacuum, as presented in Figure 2b.

The eigenmodes of this struture are calculated using the boundary element method (BEM) [34, 36, 39], see supplementary material. Finally, at this point, \( A(s, s') \) is known. Since the full EMLDOS is directly proportional to \( A(s, s') \), the EMLDOS can then be deduced. The applicability of this procedure was validated on synthetic data based on a cube in vacuum.

In order to perform the reconstruction, we used 12 spectral images (12 tilt angles), consisting in 400 * 400 spectra (see Methods). Despite the massive amount of data, the signal to noise ratio was insufficient to proceed directly to the reconstruction. Data treatment using the non-negative matrix factorization (NMF), following the mode extraction performed in the pioneer work on 3D non-vectorial surface plasmon reconstruction ([26] and Methods), leads to the modal signatures as shown in Figure 3a. The NMF spectra are consisting of several peaks, with the most prominent ones corresponding to the I/II/III modes. These features are fitted against the EELS expression as a function of \( A(s, s') \) to reconstruct the surface phonon EMLDOS as displayed in Figure 3c, and shown for a variety of directions in the supplementary material. To assure data integrity, modelled re-projected 2D EELS maps calculated from the final reconstruction were directly compared to the experimental ones, and shows good agreement (see supplementary information). With this reconstruction, mode 1 can be easily interpreted as a mode whose EMLDOS is mainly localized at the corner ("corner mode"), agreeing with previous experimental [6] and theoretical works [9]. It gets severely damped close to the substrate, similarly to what happens for corner plasmonic modes of cubes deposited on a surface [40]. The effect of the near-field environment modification on the phonon properties, predicted in [9] is revealed here for the first time. In passing, we note that, although the reconstruction basis is that of a perfectly symmetric cube in vacuum, the final reconstruction, which combines several eigenvectors,
Figure 3. Three dimensional, fully vectorial reconstruction of the phononic electromagnetic local density of state. a. NMF component extracted from the experimental data. b. Reconstructed NMF maps for the 3 modes at the two angles shown in Fig. 2. c. Three dimensional reconstruction of the EMLDOS seen from the top (substrate not shown is at the bottom of the cube). The needles indicate the direction of the polarization of the EMLDOS, while the color indicates its intensity, from red to yellow.

...does nicely reproduce the asymmetry in the corner mode. In addition, some intensity can be seen near the center of some edges. This is because the reconstruction includes both resonant (corner-localized [9]) and non-resonant (edge-localized [9]) contributions from the different modes. This points to the fact that the NMF does not provide a pure orthogonal decomposition of the different modes. It is remarkable that our reconstruction scheme allows to observe this mixing although the EELS maps and the re-projection do not. This emphasizes the need of reconstructing a physical observable, the full EMLDOS, that contains the whole physical content of a peculiar system, in contrast to the EELS data, which cannot always be directly interpreted [41]. The mode II is localized at the edges ("edge mode"), having been predicted of being a series of individual modes [9], which however could be neither measured nor identified previously. The reason is twofold: the energy shift between the corner and edge mode is extremely small, and the vectorial and spatial structure of the mode makes it difficult to probe it in the most common untitled projection geometry. Indeed, the electron only couples to eigenmodes if the electrical field is oriented along the beam direction. Also, experimental EELS signal is only accessible for non-penetrating geometries: the thickness of the cube results in a deflection of the electrons away from the spectrometer entrance aperture, as commonly encountered for thick samples in EELS. We see (see also supplementary figure 5) that the portion of the field coupled to the electron outside of the cube becomes increasingly higher as the tilt angle augments for mode II. The situation is opposite for mode III. This mode can be related to surface modes (see fig. 3c). We finally note that the effect of the substrate leads to severe damping of the three modes in its vicinity, which can be attributed to the dissipation induced by the carbon layer. Supplementary data show...
that the damping is less severe in the absence of a carbon layer.

In conclusion, this first proof of principle visualization of the surface phonon EMLDOS should motivate the development of more systematic reconstruction of the full surface phonon optical response. Such mapping should be extended to other situations where the three dimensional and vectorial information of the electromagnetic density is of importance. This includes extension of the methodology to anisotropic materials such as graphene analogues and transition metal dichalcogenides. This also includes the possibility to study consistently strong coupling physics, which has recently been unravelled for plasmons and phonons in EELS [14]. Finally, highly monochromated EELS has triggered much hope for its potential applications in vibrational mapping for biological systems [15]. However, it is well-known that the 3D information is mandatory for this purpose [42]. Therefore, the present method should be adapted to cryomicroscopy to e.g. make it possible to combine ultrastructure characterization with protein vibrational marking in 3D.

METHODS

A. Sample preparation

Samples were prepared by collecting the fumes from an ignited strip of Mg on a standard TEM grid. We tried different TEM grids, lacy carbon grids (Agar, 300 mesh Cu grid), small SiC grids), small different TEM grids, lacey carbon grids (Agar, 300 mesh ignited strip of Mg on a standard TEM grid. We tried characterization with protein vibrational marking in 3D.

B. Experiments

Experiments were performed on a modified NION HERMES-200 S at 60 keV. The modification of the microscope includes a larger pole-piece gap (6 mm) which allows for almost arbitrary tilts. The EELS spectrometer is the NION-IRIS one, fitted with a Princeton instrument KURO scCMOS camera. We used 10 mrd incidence and 15 mrd collection angles. The current was few pA and the spectral resolution in the 8-9 meV range. For each nanoparticle, a series of SI for typically 12 different angles are taken, with angles ranging from +1200 mrd to -1200 mrd (≈ ±68°). Each spectral image was constituted by 400×400 pixels. The typical acquisition time for one spectrum was 8 ms, therefore the time for a SI was around 20 min.

C. Data Analysis

Morphological reconstruction: To reconstruct the morphology of the MgO cubes, the HAADF STEM images are employed. For reconstruction we assume the sample to be a perfect cube of unknown size, electron scattering power and orientation. To find its size and orientation, model parameters are fitted to minimize the least-square error between all measured HAADF projections and projections of our model. The parameters we fit are: size of the cube, grey value (scattering power) of voxels within the cube, three angles determining the orientation of the cube and one angle determining the orientation of the tilt axis. For taking into account lateral shifts of projections (x- and y-direction) the maximum value of a filtered cross-correlation between measured and modeled projections is calculated within the optimization for each tilt angle in order to center projections. The final parameters provide the cube size, as well as its orientation with respect to all measured projections (knowing the tilt angles at which projections were acquired). These parameters are then used in the reconstruction of the phonon fields. Supplementary material shows a comparison between measured and modeled HAADF STEM projections. The fitted edge length of the cube is 191 nm.

EELS Data Processing: All EELS spectrum images were aligned by the maximum of the zero-loss peak in each spectrum and binned by 8×8, reducing the number of pixels of each spectrum image to a pixel size of 10 nm. After binning, all spectrum images were combined in a single dataset for further data processing. Before non-negative matrix factorization (NMF), all spectra were cropped to a range between 50 meV and 100 meV, which contains all major phonon peaks while excluding most of the zero-loss peak. The data was scaled to normalize the Poisson noise and an NMF algorithm [26, 43] was applied for factorization using the Hyperspy software[44].

NMF was done for different numbers of components, where it was found that five components was a good choice. In this case three components could be attributed to surface phonon excitations, well localized in both energy and spatial location of the signals, while two components could be attributed to the tail of the zero-loss peak and other excitations (see Supplementary material for NMF factors and loadings at all tilt angles).

D. Simulation

In our simulation approach we compute the EEL spectra using the MNPBEM toolbox [34]. For the dielectric function of MgO, we use a Lorentz oscillator model, as described in [6]. The experimentally determined cube size (191 nm) and orientation was used in the simulations. The substrate was not taken into account. Resonance energies were determined by simulating spectra at specific locations (corner, edge, face). This provided...
energies of 78 meV, 85 meV and 90 meV for the corner, edge and face mode. At these energies maps with the same pixel size and sample orientation as recorded in the experiment were simulated.

E. Reconstruction

The main advantage of the present quasistatic approach compared to previous reconstruction schemes is that it authorizes to get the real eigenmodes as arbitrary hybridization of the ideal structure eigenmode, and strikingly to reconstruct the effect of the substrate, which is not explicitly considered in the evaluation of the basis (see figure 2b). On the other hand, this approach presents some limit for modes, like the face modes, which are genuinely highly degenerated (within the energy resolution of the experiment). Although for general geometry and modes, this situation is not common, the cube is a particularly complicated case, especially for the face modes [9], but even for the corner where the real eigenmode is essentially composed of two idealized eigenmodes. These degeneracies are usually lifted in the relativistic case, as they rely on quasi-normal modes computed for a given value of the energy $\hbar \omega$. In order to reproduce this degeneracy lift, a subset of modes is determined through energy filtering (see supplementary material).

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