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INFLUENCE OF BRAGG SCATTERING ON PLASMON SPECTRA OF ALUMINUM

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Abstract

Plasmon spectrometry is an important method to obtain information on many-body effects in the solid state. The plasmon halfwidth and the dispersion coefficient are well investigated for a number of materials, and compare well with quantum mechanical predictions. The excitation strength of the coherent double plasmon has been investigated to a lesser extent. Experimental results are at variance with one another and with theory. This is partly due to the plural scattering which masks the coherent double plasmon.

Accurate analysis of plasmon spectra requires not only to remove the inelastic plural processes but also to take into account the coupling between Bragg and plasmon scattering at high scattering angles. It is shown that the excitation strength of the coherent double plasmon in forward direction falls below the detection limit when this correction is applied.

Key Words: Electron energy loss spectroscopy, EELS, deconvolution, inelastic scattering, plasmons, plural scattering, Bragg scattering.

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Introduction

Inelastic interactions of a fast probe electron with a specimen, measured by electron energy loss spectrometry (EELS), provide a great deal of chemical (e.g. by use of absorption edges) and electronic (e.g. interband transitions) information [4]. The strongest inelastic process is plasmon scattering. In an excitation, the plasmon (collective mode of the conduction electrons) picks up energy E and momentum $\vec{q}$ from the probe electron. The classical excitation energy E_0 of the plasmon, its halfwidth,the coeffficient α of its dispersion relation

$$E = E_0 + \alpha q^2, \tag{1}$$

and the excitation cutoff wavenumber q_c above which Landau damping is the dominant decay mechanism for plasmons are important parameters which are usually determined from experiment and compared with predictions of either classical or quantum mechanical calculations [6]. Thus, plasmon spectroscopy is a sensitive check of our understanding of many-body effects in the solid. By means of Kramers-Kronig analysis, more direct information on the electronic structure of the specimen can be obtained [3, 5, 15].

In many cases, measurements do not well compare with one another and with quantum mechanical calculation. This is not only because models are poor but also because data processing is a formidable task. Owing to the strength of the Coulomb interaction, the probe electron scatters more than once within the specimen, preventing comparison with model calculations. For accurate analysis this multiple scattering contribution has to be removed.

Theory

Inelastic interactions of electrons with a specimen are usually described by the differential scattering cross section $\partial^3 P_{\|}/\partial^2\Omega\partial E$ which relates to the loss function $Im(1/\varepsilon)$ as [8]

$$\partial^3 P_{\|}/\partial^2\Omega\partial E = Im\left(\frac{1}{\varepsilon(\vec{q},\omega)}\right)/(e\pi a_0)^2 q^2. \tag{2}$$

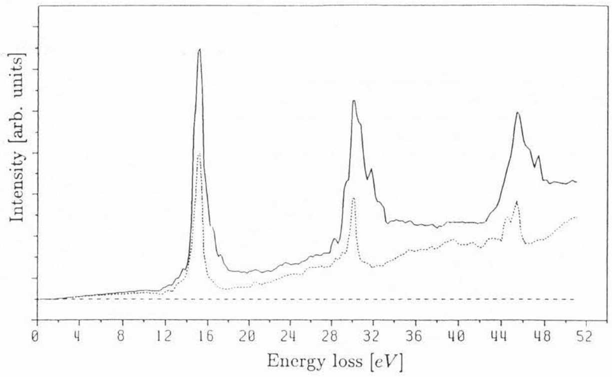

Fig. 1. Energy loss spectrum at angle $\vartheta = 19.4$ *mrad* ($q = 2.03\,\text{Å}^{-1}$): before (full line) and after Bragg-scattering correction (dashed line).

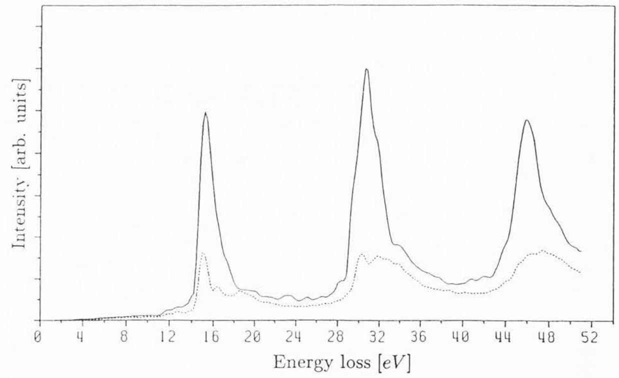

Fig. 2. The contribution of Bragg scattering to the measured profile: $\vartheta_1 = 19.4\,mrad$, ($q = 2.03\,\text{Å}^{-1}$) full line and ϑ_2=16.4 *mrad* ($q = 1.7\,\text{Å}^{-1}$), dashed line.

Here e is the elementary charge, a_0 is the Bohr radius and $q^2 = (\omega/v)^2 + k_\perp^2$. The incident electron has velocity v, the energy loss is $E = \hbar\omega$, and $k_\perp$ is related to the scattering angle ϑ as $k_\perp = k_0\vartheta$ where k_0 is the wavenumber of the incident electron. According to eq.(2), it is necessary to do angle-resolved EELS in order to obtain complete information on the scattering process. The higher the scattering angle, the more important are contributions from double and multiple inelastic scattering processes [10, 12].

At very high angles the superposition of Bragg elastic scattering has to be taken into account. In general the measured intensity can be written as

$$I_M(E,\vec{q}) = I(E,\vec{q}) * [\delta(E,\vec{0}) + \sum_{\vec{G}} \delta(E,\vec{G})] \qquad (3)$$

with the transmitted beam $\vec{0}$ and the sum over all Bragg scattered beams $\vec{G}$. For polycrystalline specimens lacking any preferential orientation of microcrystals—a situation which we shall henceforth assume—the intensities of the scattered electrons are functions of the energy loss and the wave *number* $I(E,\vec{q}) = I(E,q)$.

Let us assume for the moment that only one Bragg reflection ([111], say) is sufficiently strong to be considered. Based on a simple geometric consideration Batson and Silcox [2] give a correction of combined inelastic and Bragg scattering which shows that the intensity at a given point $\vec{q}$ is

$$I_M(E,q) = I(E,q) + \int I_B(E',q')\, I(E-E',\vec{q}' - \vec{q})dE'dq'. \qquad (4)$$

Here I_M is the measured spectrum,

$$I_B = \frac{A_B}{2\pi q_B}\delta(q' - q_B)\delta(E - E') \qquad (5)$$

is the intensity which stems from Bragg reflections (i. e. purely elastic scattering) and $I(E,q)$ is what would be measured if no Bragg reflections were present. A_B is the relative integral intensity of the Bragg ring in question.

After some elementary calculation we get

$$I(E,q) = I_M(E,q) - A_B/\pi \int_0^\pi I(E, Z(q,q_B,\varphi))d\varphi. \qquad (6)$$

The factor $Z(q,q_B,\varphi)$ is obtained from the geometry of the Bragg ($\vec{q}_B$) and scattering wave vector ($\vec{q}$) as

$$Z(q,q_B,\varphi) = \sqrt{q_B^2 + q^2 - 2q_B q\, cos\varphi}. \qquad (7)$$

Eq.(6) can be solved by iteration [2]. In the first approximation we obtain

$$I_A(E,q) = I_M(E,q) - A_B/\pi \int_0^\pi I_M(E, Z(q,q_B,\varphi))d\varphi, \qquad (8)$$

which can be considered sufficiently accurate since A_B is of the order of percents.

In the following calculations only the [111]-reflection in aluminum was considered. An accurate analysis should also include the [200] and [220] reflections; however, from the following results it will become clear that the latter do not much influence energy loss spectra up to $q = 2.7\,\text{Å}^{-1}$, the highest wave number where spectra were measured.

Experiments

The numerical calculation was performed for energy loss spectra of aluminum. The material was evaporated from a tungsten boat at 5.10^{-6} *torr* and condensed onto glass substrates covered with Mowital. The deposition rate was 1.5 nm/s, and the films were 200 nm thick. The films were floated off the substrate in Chloroform and prepared onto Cu-grids for electron microscopy as usual. Measurements were done with a cylindrical mirror analyzer attached to a modified Siemens Elmiskop IA. The electrons were accelerated to an energy of 40 keV. The energy resolution was typically $0.75\,eV\,(FWHM)$. In the diffraction mode, energy scans were performed with an angular resolution of 0.17 *mrad* to 0.8 *mrad*, depending on scattering angle.

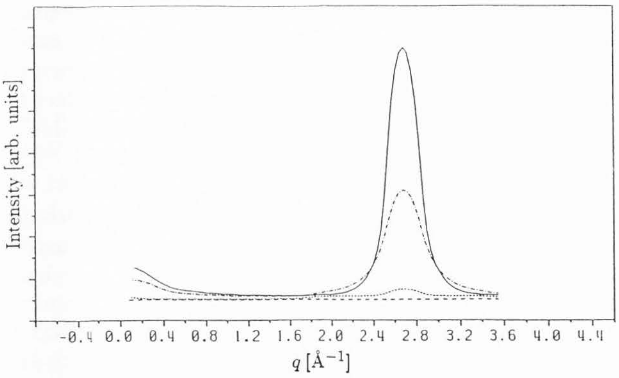

Fig. 3. The contribution of Bragg scattering as a function of momentum transfer. Full line: $E_1 = 15\,eV$, first plasmon; dashed line: $E_2 = 17.2\,eV$; dash-dotted line: $E_3 = 30\,eV$, second plasmon. The maximum is located at $2.7\,\text{Å}^{-1}$, corresponding to the Bragg angle for (111)-reflections, $\vartheta = 26.6\,mrad$.

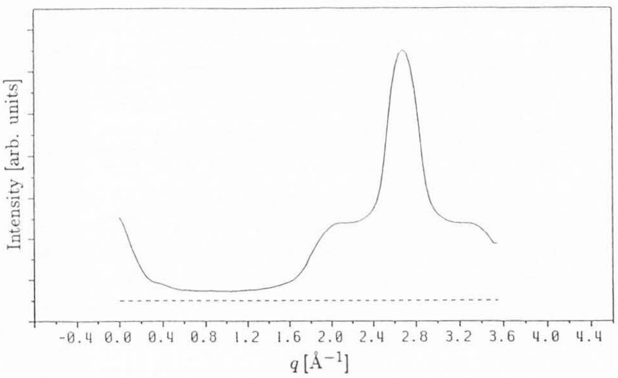

Fig. 4. Detail from Fig.3. ($E = 17.2\,eV$).

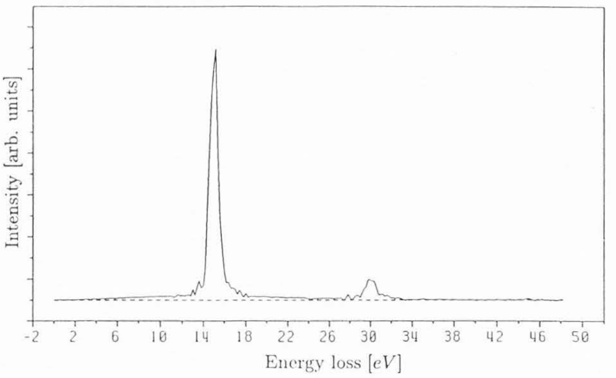

Fig. 5. Single loss spectrum at small scattering angle $\vartheta = 0.34\,mrad$ ($q = 0.035\,\text{Å}^{-1}$), obtained by matrix-deconvolution.

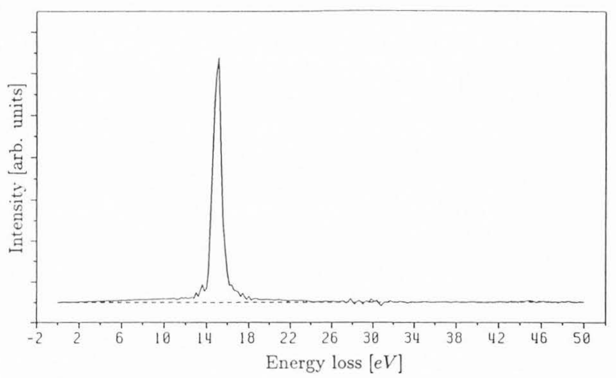

Fig. 6. The same spectrum as in Fig. 5 with Bragg scattering correction applied prior to deconvolution.

Results

In Fig.1 the measured (full line) and the corrected (dashed line) spectra at scattering angle $\vartheta = 19.4\,mrad$ ($q = 2.03\,\text{Å}^{-1}$) are compared. Note the overall increase of intensity with energy loss; a tendency especially found at high scattering angles. It should be mentioned at this point that we used as-measured spectra—no smoothing procedure was applied. Fig.2 shows the contribution of Bragg scattering (difference of graphs of Fig. 1) at two scattering angles, ϑ_1=19.4 $mrad$ ($q = 2.03\,\text{Å}^{-1}$), full line and ϑ_2=16.4 $mrad$ ($q = 1.7\,\text{Å}^{-1}$), dashed line. The asymmetric shape of the plasmon-like single, double and triple excitation peaks is due to the fact that the Bragg ring acts as a source now instead of the incident beam; thus the contribution of plasmon excitation with non-vanishing wave vector is increased as can be easily imagined from the scattering geometry. The smaller intensity at $n \cdot 15\,eV$ where $n = 1, 2, 3$ for $16.4\,mrad$ is caused by the strong decrease of intensity at these energies for higher scattering angle, with the consequence of stronger asymmetry. From the figure, it is obvious that the Bragg correction becomes more important with increased scattering angle. Fig. 3 shows the Bragg contribution as a function of momentum transfer in the diffraction plane at different energy loss ($E_1 = 15\,eV$, first plasmon; $E_2 = 17.2\,eV$,dashed line; $E_3 = 30\,eV$, second plasmon, dash-dotted line). The correction is important only in the vicinity of the Al (111) Bragg ring at $q = 2.7\,\text{Å}^{-1}$ ($\vartheta = 26.6\,mrad$). Fig. 4 is a detail from the dotted line in Fig. 3. (Energy loss $17.2\,eV$). The plateau between 1.8 and 3.6 Å^{-1} is caused by plasmon dispersion: at $q = 0.92\,\text{Å}^{-1}$ the plasmon has an energy of $\sim 17.2\,eV$. Hence, at $17.2\,eV$ energy loss, and as a function of momentum transfer, considerable intensity can be expected in a distance of $q = 0.92\,\text{Å}^{-1}$ away from the Bragg ring.

Figs. 5, 6 demonstrate the effect and magnitude of the Bragg correction to angle resolved plasmon spectra. Fig. 5 is a single loss spectrum at 0.34 mrad ($q = 0.035\,\text{Å}^{-1}$) obtained by a deconvolution procedure developped by one of the authors and coworkers [7, 10, 12].

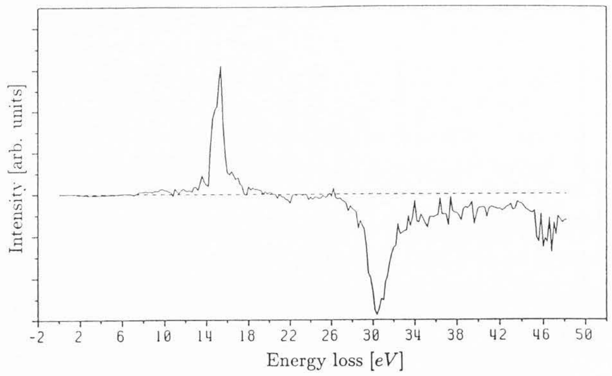

Fig. 7. Single loss spectrum at large scattering angle $\vartheta = 19.4\,mrad$ ($q = 2.03\,\text{Å}^{-1}$) , obtained by matrix-deconvolution.

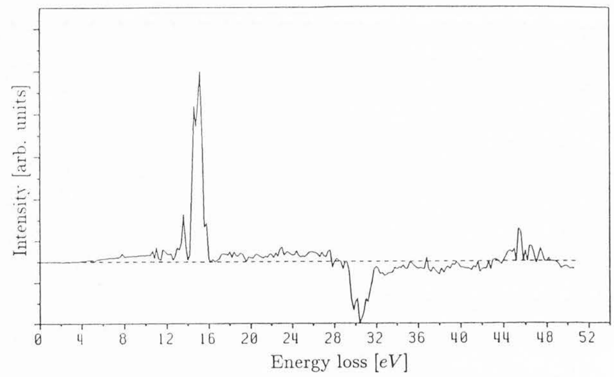

Fig. 8. The same spectrum as in Fig. 7 with Bragg scattering correction applied prior to deconvolution.

Note that the double plasmon loss at 30 eV has vanished (or has fallen below detectability, at least) when the Bragg scattering correction is applied (Fig. 6). Up to now it has been thought that the Bragg elastic contribution to plasmon spectra was negligibly small at that small scattering angle; consequently, the subsidiary maximum at the double plasmon energy has been interpreted as a coherent excitation of two plamons in one scattering process [9, 11, 13]. Fig. 7 is the same for a scattering angle of 19.4 mrad ($q = 2.03\,\text{Å}^{-1}$). Despite the fact that the statistical scatter of data is much larger at high angles, the improvement of Fig. 8 over Fig. 7 is evident. The small negative dip at $30\,eV$ is caused by "hyper-deconvolution" of the double plasmon maximum, but apparently the effect is partly due to the Bragg scattering contribution.

Conclusions

The effect of superposition of elastic (Bragg) scattering on angle resolved plasmon spectra of aluminum was investigated. We find that the Bragg contribution is largest at high angles as was to be expected. An appropriate correction prior to a multiple scattering deconvolution yields smaller negative dips at the double plasmon for high scattering angles, and removes the double plasmon at low scattering angles almost completely. For accurate analysis of energy loss spectra in the medium loss range, such as determination of the dielectric function for non-vanishing wave number, it is important not to neglect the Bragg elastic superposition.

Acknowledgements

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pertes d'électrons rapides diffusés dans la direction du faisceau incident. J. de Physique 36, 199-213.

Discussion with Reviewers:

C. Colliex: Why was a 200 nm thick Al specimen used for 40 keV? The mean free path Λ_p for inelastic scattering is then rather short compared to thickness t. What is t/Λ_p in your experiment?
Authors: We decided to use a thick specimen ($t/\Lambda_p \approx 4.4$) because multiple scattering is then prominent. Thick specimens impose a difficult test upon multiple scattering removal. Usual Fourier deconvolution would no more work at that thickness as was recently shown [14]. Therefore, this experiment is also a demonstration of the validity and accuracy of the algebraic matrix deconvolution developed by two of the authors [7, 10, 12].

C. Colliex: There is a serious omission for a clear understanding of your paper, i.e. a chart of the investigated modes on a $(E, \vec{q})$-graph. Some of your figures deal with sections along a given q value (Figures 1 and 2), and others along a given E value (Figures 3 and 4). Considering the first set of data, can you explain the overall increase of intensity with energy loss, is it due to electron-hole pair excitations?
R. F. Egerton: You state that the higher the scattering angle, the more important are contributions from plural scattering; is this because of the increased angular width of the plural scattering angular distributions compared to the single-scattering distribution?
Authors: We do not believe that an (E, q)-graph is useful to the reader. The graph C. Colliex refers to would show the same modes as already discussed in [2], a paper explicitely aimed at determination of the low loss function in Al, with emphasis on plasmon dispersion. The present paper however aims at a totally different issue, *viz.* showing the influence of Bragg scattering on deconvolution, with emphasis on the residual double plasmon intensity. This is most clearly displayed as an energy scan at fixed momentum transfer, as given in Fig. 2, for instance. Contrary to [2], we use a more accurate method for multiple scattering deconvolution, and we come to a different conclusion on the magnitude of the coherent double plasmon excitation.

The answer to R. F. Egertons question is yes. In Fig. 1, electron-hole pair excitations are so seriously masked by multiple scattering that they are not visible. The faint positive background in Fig. 8 is due to those excitations.

C. Colliex: Are the satellite peaks on the right sides of the double and triple plasmon peaks real features or noise? If they are real features, I do not understand why they disappear after Bragg removal. In a connected domain, you plot the Bragg contribution in Figure 2. Could you comment about the dissymmetric shape, the exact energy position of the rises, the satellites at higher energy? When you plot the results along the q scale, you show that for the plasmon at $E_1 = 15\,eV$, the Bragg contribution dominates at the Bragg angle and that there is no plateau around the peak, it seems to be the result of the full line curve in Figure 3. But for Figure 4 which deals with the $17.2\,eV$ loss (i.e. a plasmon with $q = 0.8\,\text{Å}^{-1}$) I understand the origin of the plateau but I miss your argument about the main maximum which is from the tail of the plasmon peak at $q = 0.0\,\text{Å}^{-1}$. Why does it come at $17.2\,eV$ and $2.7\,\text{Å}^{-1}$?
Authors: The satellite peaks, or rather the shallower slopes of the high-energy sides of the plasmon peaks in Fig. 1 are contributions from energy shifted plasmons, emerging from the (111) Bragg ring. This asymmetry is removed by the Bragg correction. As is evident from Fig. 2 the Bragg contribution is more asymmetric for smaller scattering angle, and the center of gravity is shifted to the right. This is because a smaller scattering angle implies *higher* scattering angle with respect to inelastic events emerging from the Bragg ring. The spectrometer aperture positioned at $19.4\,mrad$ receives inelastic intensity from the Bragg (111) ring scattered by a minimum of $5.8\,mrad$. At this angle the triple plasmon excitation maximum is about $46\,eV$ which is exactly the position of the third plasmon maximum found in Fig. 2, full line. For the dashed line, this estimate gives a value of $48\,eV$, again corresponding with Fig. 2. The low energy satellites at $15\,eV$ and $30\,eV$ are undispersed plasmon peaks caused by diffuse elastic and quasielastic scattering.

The main maximum at $q = 2.7\,\text{Å}^{-1}$ (corresponding to the Bragg angle for (111) reflections) comes at any energy loss. It is caused by the fact that at the Bragg ring q_{Bragg}, the spectrometer aperture receives inelastic intensity from the Bragg (111) ring scattered by a minimum of $0\,mrad$. The onset of the superimposed plateau which is more or less symmetric with respect to q_{Bragg} depends on energy loss E and is given by

$$q_P = q_{Bragg} - q_E.$$

In Fig. 4, $E = 17.2\,eV$, $q_E = 0.92\,\text{Å}^{-1}$, and $q_P = 1.78\,\text{Å}^{-1}$.

C. Colliex: Why would there remain a contribution at $30\,eV$ due to elastic Bragg scattering?
Authors: The Bragg correction reduces intensity in a nontrivial manner as discussed above. So, it is difficult to decide a priori which effect the correction will have on the outcome of the deconvolution. Basically, the angular halfwidths of the single, double and triple plasmon profile are changed in different ways by combined inelastic and Bragg scattering such that the deconvolution produces artifacts without Bragg correction - see the remaining negative intensity at $19.4\,mrad$ in Fig. 7. We cannot really answer this question—in our opinion the correction does.

P. Batson: Your conclusion leads one to believe that all previous work on double plasmon scattering is contaminated by the Bragg scattering problem. The work in ref. 2 was done on (111) epitaxial films. Therefore, the (111)

and (200) type reflections were absent. The (220) reflection at 4.4 $\mathring{A}^{-1}$ was the only Bragg scattering that needed to be treated. The work in reference 13 was done in single crystal Al oriented to minimize Bragg scattering.
Authors: Previous work on double plasmon scattering did not even agree on the order of magnitude of the coherent double plasmon, experimental values ranging from 0.5% to 13%—see the review in reference [9]. The theory of Ashley and Ritchie [1] for the two-plasmon intensity is very sensitive to the choice of the cutoff wavenumber, resulting in a range of possible values from 4% to 17% in Al. So, there seems to be a problem with the early experiments and with calculations.

The work in reference [2] probably was contaminated by Bragg scattering for wavenumbers less than $\sim 1.4\,\mathring{A}^{-1}$ because the correction—according to Eq. 12 in reference [2]—is effective only beyond this value. Furthermore, our deconvolution routine uses a spline approximation to a Lorentzian whereas the deconvolution routine used in reference [2] relied on a Gaussian fit to the angular profiles, possibly causing convergence problems.

P. Batson: The angular transform was exact. The Gaussian fit was used to reduce the dynamic range of the data for the numerical Fourier Bessel Transform. The resulting transform was the sum of the analytical transform of the Gaussian plus the numerical transform of the residual obtained after subtraction of the fitted Gaussian.
Authors: When you transform the residual you need an infinitely large base interval because the experimental profile falls to zero at a finite angle whereas the Gaussian does not. It is not clear from reference [2] how you cope with this problem numerically.

To clarify our point: We do not state that reference [2] is erroneous. We simply say that one cannot avoid approximations in data analysis, and that the approximations inherent in the present work were different from those in reference [2] as stated above.

It is not the scope of the present paper to judge whether or not these differences in data analysis can explain the difference in the coherent double plasmon intensity given in reference [2] to the present one (which is beyond detection limit). We trust in our results not only because the deconvolution routine used in the present work is known to be highly successful—see reference [11]—but also because the Bragg scattering would tend to increase the two-plasmon intensity with specimen thickness. This behaviour has been observed—see the following discussion with R. F. Egerton.

P. Batson: Quite frankly, I believe that if the double plasmon is not the answer, then there must be a subtle piece of physics operating that remains unappreciated. In this context, I find myself intrigued by the effect of the Bragg correction. If the obviously-present (111) Bragg scattering in this experiment gives such a precise correction at the double plasmon position, perhaps something similar happens when the Bragg scattering is not so obvious. A coherent plasmon-Bragg event (an Umklapp process) would satisfy the need. Then the plasmon plus Bragg scattering need not be confined to the normal Ewald sphere. In spite of the second order nature of this process, under dynamical scattering conditions this may be possible.
Authors: This is an attractive idea, and we feel that it could explain why Spence and Spargo [13] who used a single crystal of aluminum came up with such high a value for the double plasmon intensity. An answer to this question would however require a systematic investigation of the double plasmon under dynamical as well as kinematic scattering conditions.

Apart form that, two other facts might be responsible for the extremely high value (twice as much as the value reported in reference [2]) given in reference [13] for the two-plasmon intensity: 1) The results of this method will depend on the choice of the energy window; 2) Fig. 3 in reference [13] shows considerable and systematic difference between measurement and the best fit. In our opinion, the measurements suggest a constant additive background superimposed on the Poissonian distribution, and this is certainly not explained by the hypothesis of a coherent double plasmon.

R. F. Egerton: Is the contribution near the origin in Fig. 3 due to double Bragg scattering back to $\theta = 0$? If so, it presumably requires no "correction" in the sense that double scattering increases the zero-loss and plasmon-loss intensities by the same fraction.
Authors: According to eq. 8, the correction term is calculated from the measured spectrum which has a subsidiary maximum at θ_{Bragg}. This will cause a subsidiary maximum in the correction term at $\theta = 0$. Literally, this is double Bragg scattering back to the origin—albeit slightly overestimating the correction since eq. 8 is approximate. Your conjecture is probably right for $\theta = 0$.

R. F. Egerton: Presumably your factor Z in Eq. 6 takes into account the angular distribution of plasmon scattering? Do you allow for a cutoff at some critical wavevector?
Authors: The factor Z in Eqs. 6 and 8 is the distance of the spectrometer aperture in the diffraction plane from the Bragg ring, as a function of the azimuthal angle φ. The angular distribution of the plasmon intensity is the measured intensity. Since we measured up to $q = 3.5\,\mathring{A}^{-1}$ this is the cutoff wavevector for the Bragg correction.

R. F. Egerton: Is it correct to say that the Bragg correction increases with increasing specimen thickness, and that the 30 eV artifact seen in Fig. 5 would therefore be considerably smaller for a 50 nm (as opposed to 200 nm) specimen?
Authors: This is plausible although we would not expect a linear increase of the artifact. There is in fact experimental evidence for a thickness dependence. Schattschneider and Pongratz [9] found the remaining intensity at 30 eV in Aluminum to be thickness dependent after deconvolution of image mode spectra ($\sim$ 3% of single loss for a 200 nm thick specimen, $\sim$ 1% for 50 nm thickness). There was no Bragg correction applied in that work.