Plasmon behavior of Zn from electron-energy-loss spectroscopy

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The momentum-dependent dielectric function of Zn was measured using electron-energy-loss spectroscopy (EELS) in transmission. The loss function in the low-energy region is found to be a superposition of a sharp absorption feature due to the excitation of electrons from the $3d$ level at a binding energy of 9.4 eV and a broad not well pronounced feature at about 14 eV, which is attributed to the collective plasmon excitation of the free charge carriers. This latter feature becomes more pronounced with increasing momentum transfer due to a strong decrease of the core-level oscillator strength. The decrease of oscillator strength leads to an initially vanishing dispersion of the plasmon, and to a reduction of the plasmon line width with increasing momentum transfer. This behavior can qualitatively be understood in the framework of a Drude-Lorentz approach. [S0163-1829(97)06540-5]

I. INTRODUCTION

The dielectric properties of electrons in a metal are determined by the free charge carrier plasmon, interband transitions, and core-level excitations, and their mutual interactions. The excitation of plasmons in nearly free-electron systems, i.e., in simple metals, has been investigated since 1950.[1] In the beginning, interest was focused on the influence of the band structure on the plasmon properties. An extensive overview of these influences was given by Sturm.[2] Later, the influence of correlation and exchange effects was investigated by studying deviations of the plasmon dispersion from mean-field theories, such as the random-phase approximation (RPA).[3] As a result it was found that the lowering of the dispersion coefficient compared to RPA values could quantitatively be described by local-field corrections in the case of simple metals.[4] In the case of heavy alkali metals, however, where exchange and correlation effects should be most pronounced, a negligible and even negative dispersion was found for Rb and Cs, respectively.[5] These findings have stimulated much discussion and controversy, up to the present time.[6]

In most simple metals, plasmons and core-level excitations are well separated ($\hbar\omega_p \ll E_{\text{core}}$). The polarization of the cores is taken into account by introducing an energy- and momentum-independent dielectric constant, which shifts the free-electron plasmon energy to lower values, but does not influence its dispersion. In the case of the post-transition metals, systems with shallow core levels having binding energies comparable to the plasmon energy can be found. In these cases, a strong interaction between plasmons and the core levels is to be expected, which should lead to significant deviations from predictions of the nearly free-electron approach. The plasmon energy of such metals has been investigated by several techniques, in particular by electron-energy-loss spectroscopy (EELS). Most of the investigations up to now have been restricted to the long-wavelength limit, i.e., to the case of vanishing momentum transfer. It has been found that the experimental plasmon energy $\hbar\omega_p^{\text{expt}}$ is often significantly lowered[7,1] compared to the plasmon energy within a Drude-Lorentz model, in which the plasmon frequency is given by $\omega_p^D = (4\pi ne^2/m)^{1/2}$. Investigations of the momentum dependence of the dielectric properties, however, are rather scarce. The dispersion coefficients published by different authors for post-transition metals have often been found to be higher than the predicted RPA values[1,7–9]—a behavior which is in striking contrast to simple metals such as the alkali metals or Al, where dispersion coefficients have been found to be significantly smaller than the RPA predictions due to correlation and exchange effects.

In a series of publications, Sturm *et al.*[10] established a microscopic theory which explains the behavior of the dielectric properties in the long-wavelength limit for electron systems in the presence of shallow core levels. The polarization of the valence electron was calculated as for free electrons in the framework of the RPA, and the core electrons were taken into account via a model of finitely extended dipole moments placed on a cubic lattice, comparable to a Clausius-Mossotti ansatz. The microscopic reason for the redshift of the plasmon energy with respect to the conventional Drude value was found to be not only a simple screening due to the dielectric background, like in a conventional Drude-Lorentz approach as described above, but also to involve a subtle interplay between an upward shift, due to an increase of the effective electron density and a decrease due to the polarization of the dipole lattice. The decrease overcompensates for the increase, hence leading to a net redshift of the plasmon energy. This theory also describes quantitatively the edgelike shape of the core-level excitations in the long-wavelength limit. The predictions of this theoretical approach are found to be in reasonable agreement with experimental data for Cd, In, and Sn, i.e., for metals where the core levels lie slightly above the plasmon energy. A detailed theory for the momentum dependence of the plasmons and the core polarization has not been established up to now, to our knowledge. It has only been speculated that the high dispersion coefficients found in the relevant metals could be caused by a dynamic interaction of the free carriers with the core polarization.

From this point of view, Zn is one of the most interesting

post-transition metals. In many experimental and theoretical studies it has been shown that near the Fermi surface its band structure can be described quite satisfactorily within the model of almost free electrons with a density given by two electrons per atom.[11] On the other hand, it has been shown by band-structure calculations that the lower part of the conduction band is intersected by the $3d$ bands.[12] This brings into question the classification of the d electrons as being tightly bound, i.e., as belonging to individual cores. Experimental evidence for the delocalized nature of d electrons was given by Himpsel *et al.*, who showed, with angular-dependent photoemission spectroscopy, a dispersion of the $3d$ band along the hexagonal axis.[13] The intersection of the $3d$ bands with the conduction band should lead to a strong dynamic interaction between the free charge-carrier plasmon and the core-level excitations, and hence to significant deviations from a description of collective processes within a free-electron model. Though there are many interesting effects to be expected, the experimental situation regarding the dielectric response of Zn is still not clear. Up to now there is no consensus in the literature as to whether a collective plasmon excitation of the free charge carriers can be observed in Zn or not, even in the long-wavelength limit. In recent decades, plasmon energies for Zn between 9 and 17 eV have been reported.[8,14–19] To our knowledge, no momentum-dependent investigations can be found in the literature up to now. Therefore it seems desirable to us to investigate the momentum dependent dielectric response of Zn in detail. A method well suited for such an investigation is EELS in transmission. The cross section for inelastic electron scattering is proportional to the so-called loss-function $\mathrm{Im}[-1/\epsilon(q,\omega)]$. In this paper we present results from such EELS investigations performed on free-standing polycrystalline Zn films.

II. EXPERIMENT

Polycrystalline Zn films with a thickness of about 1000 Å were prepared by evaporating high purity metal (99.999%) under high-vacuum conditions. In order to avoid oxidation of the highly reactive films and to prepare free-standing samples without any substrate, we followed a method outlined by Imbusch[20] for the preparation of thin Pb films. As substrate for the film preparation, frozen methanol on a fine-mesh copper grid at a temperature of about 100 K was used. To remove the methanol, the samples were carefully heated to the melting point of the methanol (180 K), and kept at this temperature until the methanol evaporated. The low temperature prevents any degradation of the films. Subsequently, the films were quickly transferred into the spectrometer after the temperature had been raised to room temperature. The phase purity of the films was checked by elastic electron scattering where no signs of other phases, in particular ZnO, could be detected. A very thin unavoidable surface oxide layer is of negligible importance, as it does not influence the bulk properties and leads only to minor surface contributions in the EELS spectra, which were taken in transmission geometry.

The measurements were performed with a spectrometer described in detail elsewhere.[21] The primary electron-beam energy was set to 170 keV. The energy and momentum resolution were chosen to be 110 meV and 0.05 Å^{-1}, respectively.

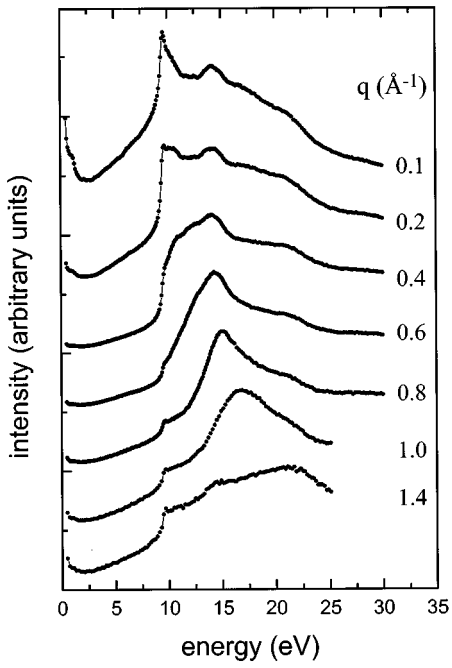

FIG. 1. Electron-energy-loss spectra of Zn for different momentum transfers q.

The spectrum of hcp Zn is not isotropic, even in the long-wavelength limit. The spectra measured from a polycrystalline sample are therefore the result of an averaging over all spatial orientations of the wave vector **q**. However, it is generally accepted that the anisotropy of the plasmon properties is rather small in pure metals,[1] so that the averaging only leads to an additional small broadening of the present structures. A detailed estimate of the expected anisotropy in Zn can be found in Ref. 8, where a difference of the plasmon positions parallel and perpendicular to a hexagonal axis of less than 0.2 eV has been calculated.

III. RESULTS AND DISCUSSION

In Fig. 1 we show selected EELS spectra of Zn for different momentum transfers q. Let us start with the discussion of the lowest momentum-transfer spectrum $q=0.1$ Å^{-1}. It differs significantly from what is expected for a simple free-electron metal. It is dominated by a narrow and sharp absorption feature at about 9.4 eV. Its steplike behavior can clearly be seen by differentiation of the loss function, which leads to a Gaussian-shaped peak centered at 9.38 eV with a full width at half maximum of 0.11 eV, i.e., it is as narrow as the energy distribution of the incoming beam. Comparing the measurements of several films with different thickness (500–1000 Å), we exclude the possibility that this structure is related to a surface excitation. The thinner films show a weak additional surface contribution at about 6 eV, which was identified as the surface plasmon by the **q** dependence of the scattering cross section.[1] In Ref. 8 a qualitatively similar spectrum of Zn for $q=0$ has been reported, but the lower resolution therein smeared out the sharp peak of the absorption structure. The thresholdlike behavior of the structure suggests an interpretation involving an excitation from a shallow core-level to the conduction band. The shoulder on

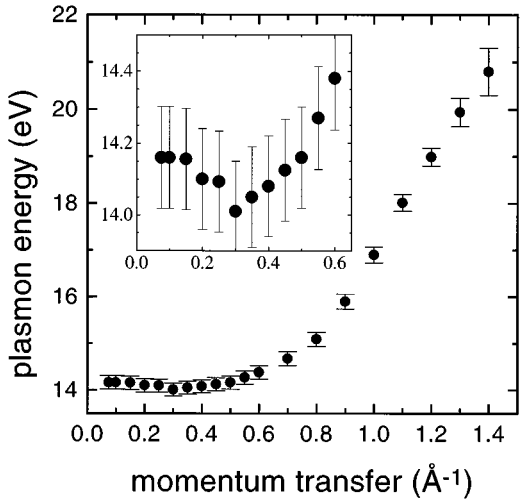

FIG. 2. Dispersion of the charge-carrier plasmon. The inset shows data for low momentum transfer on an expanded scale.

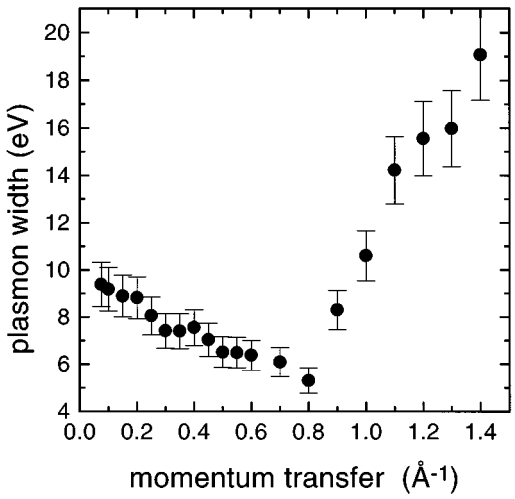

FIG. 3. Full width at half maximum of the plasmon line vs the momentum transfer q. These values have been obtained by fitting a symmetric Lorentzian to the plasmon peak as described in the text.

the high-energy side of this absorption peak results probably from slightly deeper-lying d bands. It turns into an individual peak with increasing momentum transfer. The determination of the onset energy for the second absorption structure cannot be done in the same exact way as for the first one. When taking the turning point directly after the sharp absorption feature, a value of 10 eV is derived.

The values of the two core-level excitation energies can be compared with binding energies of the $3d$ bands, which have been determined by x-ray photoelectron spectroscopy.[22] There, a value of 9.40 eV was found for the Zn $3d$ band, and a second peak at 9.95 eV, which is attributed to the spin-orbit split-off band. These results agree very nicely with the excitation energies derived in this work.

Besides the features in the loss function which were attributed above to core-level excitations, a relatively wide but nevertheless well-pronounced structure can be observed at about 14.2 eV. This value is only slightly higher than the Drude plasmon energy of 13.5 eV. So it seems straightforward to identify this feature as the volume plasmon of the free charge carriers. A discussion of the momentum dependence of the structure will confirm this interpretation. A comparable plasmon feature is also found in the work of Gorobchenkov *et al.*,[8] while in other experimental work it has not been detected or discussed. For higher energies ($20\ \text{eV} \leqslant \hbar\omega \leqslant 30\ \text{eV}$) the intensity of the loss function decreases. In the measured energy range, no sharp and well-defined additional features appear. The energy losses are mainly caused by double-scattering processes which become more and more important for higher momentum transfers. At very low energies an increase of intensity with decreasing energy can be observed, which is due to quasielastic scattered electrons. The weak structure at about 1.5 eV is most likely caused by low-lying interband transitions, as can be argued when taking into account the band structure calculations[12] for Zn. Such interband transitions are of minor importance for the topic of the present work due to their small oscillator strength.

When the momentum transfer is increased, the spectrum successively changes its shape. Qualitatively it can be seen that the core-level excitation intensity decreases dramatically and reappears for higher momentum transfers. The position of the volume plasmon stays nearly constant at about 14 eV, but shifts to higher energies when the momentum transfer is increased above $0.5\ \text{Å}^{-1}$. Additionally, the plasmon feature becomes more pronounced, and dominates the spectrum for intermediate values of q. For momentum transfers higher than 0.8Å^{-1}, the plasmon significantly broadens. It disappears in the background of multiple-scattering processes at momentum transfers higher than $1.4\ \text{Å}^{-1}$. Additionally, with the reappearance of the absorption edge in the high momentum spectra, a weak, plasmonlike feature appeares at 14 eV. This can be explained by a double-scattering process which is a well-known effect in EELS measurements: elastic scattering of the electrons by imperfections or by phonons involving a change of direction and additional inelastic scattering without considerable momentum transfer. Such double-scattering processes become more and more important for higher momentum-transfer spectra, and hence lead to these additional structures.

Let us consider the position and the shape of the volume plasmon in more detail. The position of its peak as a function of the momentum transfer, i.e., its dispersion, is shown in Fig. 2. The position of the plasmon was determined by fitting a Lorentzian to the spectral region of the plasmon. Because the plasmon excitation is the dominant absorption process in metals in this energy range, we fitted this curve without any background within the energy interval where the plasmon shows its peak structure. The low momentum-transfer region of the dispersion is shown in the inset of Fig. 2 in an expanded scale. Here it can be seen that there is a vanishing or even a slightly negative dispersion for momentum tranfers up to $0.3\ \text{Å}^{-1}$. Then the dispersion becomes slightly positive, but it is not before $0.5\ \text{Å}^{-1}$ that the plasmon energy significantly differs from 14.2 eV. The full width at half maximum (FWHM) of the Lorentzian, which we used for the evaluation of the plasmon peak, decreases with increasing momentum transfers from about 9 eV at $0.08\ \text{Å}^{-1}$ to 5 eV at $0.8\ \text{Å}^{-1}$, as can be seen in Fig. 3. At higher momentum

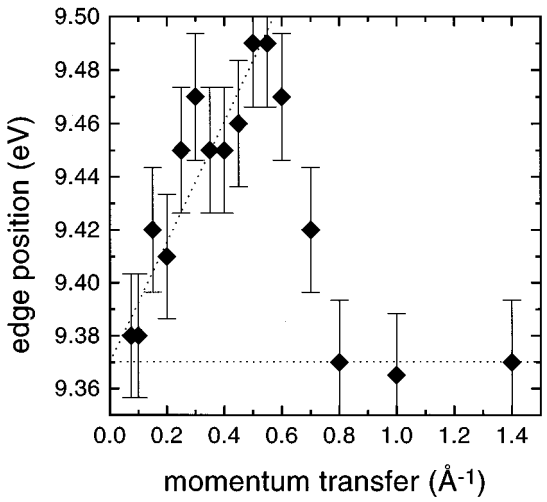

FIG. 4. Energy position of the absorption onset of the $3d$ core-level excitations.

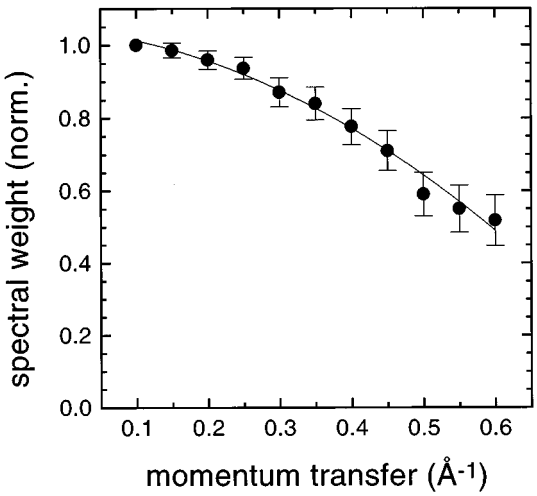

FIG. 5. Oscillator strength of the core-level excitations as determined via the sum rule as a function of the momentum transfer q. The values are normalized to the oscillator strength at $q = 0.1$ Å^{-1}.

transfers the width of the curve rapidly increases, a behavior which is attributed to the onset of the strong decay of the plasmon. It should be remarked that the determination of the FWHM of the loss peak is rather problematic for such a broad structure as the plasmon in Zn. The influence of other structures in the direct vicinity cannot be fully taken into account. Thus the absolute values must be interpreted with care because it is very likely that we have overestimated the intrinsic linewidth to some degree. Nevertheless, it can be stated that the plasmon narrows with increasing momentum transfer up to 0.8 Å^{-1}.

The sharpening of the plasmon line with increasing momentum transfers is quite unusual, because usually a quadratic increase of the linewidth with increasing momentum transfer is observed in metals,[1] with the exception of Li, where a sharpening of the plasmon line with increasing momentum transfer has also been observed.[5]

For a better understanding of the unusual dispersion and behavior of the plasmon line shape we have to consider the core-level excitations, which also show an unusual behavior when the momentum transfer is increased. Figure 4 shows the onset of the core-level absorption as determined from the turning point of the steplike feature with increasing momentum transfer. It can clearly be seen that a slight shift to higher energies takes place when the momentum transfer is increased up to 0.6 Å^{-1}. Then the onset energy apruptly returns to its original value. This behavior can be explained by the double-scattering process described above. So we can argue that for momentum transfers higher than 0.6 Å^{-1} contribution from the spectrum at $q=0$ are significant and cannot be separated. Hence we will restrict ourselves to $q \leqslant 0.6$ Å^{-1}.

For a quantitative estimation of the decrease in intensity of the core-level excitations, we calculated the effective electron number N_{eff} by applying the sum rule $N_{\mathrm{eff}} \sim \int \omega \ \mathrm{Im}[-1/\epsilon(\omega)]d\omega$ to the energy region where these excitations take place, i.e., from 9.4 to 11.4 eV. The spectra were normalized using the well-known decrease of the cross section of inelastic electron scattering,[1] which varies as $1/q^2$. The calculation of the effective electron number was only performed for momentum transfers up to 0.6 Å^{-1} in order to avoid contributions from the double-scattering processes described above. Figure 5 shows the effective electron number, or equivalently, the oscillator strength, as determined by the above given sum rule. The values are normalised to the $q = 0.1$ Å^{-1} oscillator strength. The error bars result from the uncertainties in the normalization procedure. A quadratic decrease of the effective electron number to about 50% of the $q = 0.1$ Å^{-1} value can be detected. In order to determine the influence of this decreasing oscillator strength on the plasmon behavior, we performed a model calculation within a modified Drude-Lorentz model. As outlined in Ref. 8, the absorption of the core-level excitations cannot be described by a simple Lorentz oscillator model alone, since the transitions take place into the energetically wide conduction band starting from a threshold energy E_d. A more appropriate description can be given by an infinite sum of Lorentzians:

$$\epsilon_d(\omega) = \sum_k \frac{f_k}{\omega_k^2 - \omega^2 - i\gamma_k\omega}. \tag{1}$$

In our case the values of the eigenfrequencies vary continuously from E_d to ∞, so that we can convert from a summation in k to an integration with respect to ω_k. Furthermore we assume the damping parameters γ_k to be k independent (γ), and the oscillator strength to be given by the relation $f_k \sim 1/\omega_k^2$. Then ϵ_d becomes

$$\epsilon_d(\omega) = f_d E_d^2 \int_{E_d}^{\infty} \frac{dx}{x^3(x^2 - \omega^2 - i\gamma\omega)}, \tag{2}$$

where f_d is a measure of the total oscillator strength of the core-level excitations. The above given integral can be evaluated analytically, hence leading to the complete dielectric function

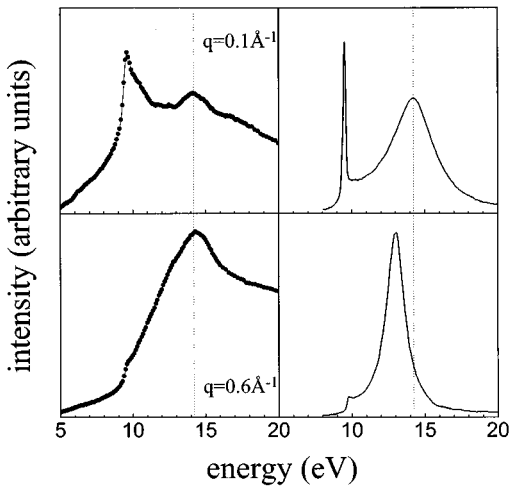

FIG. 6. Comparison of experimental spectra (left side) and model calculations (right side) based on a modified Drude-Lorentz model as described in the text. The dotted line gives the energy position of the $q=0.1$ Å^{-1} plasmon at 14.2 eV.

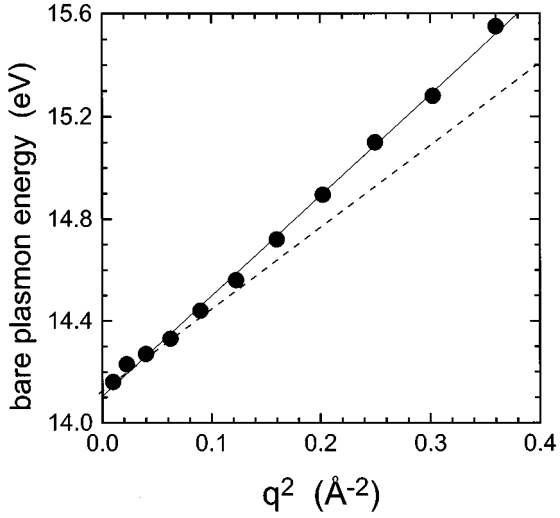

FIG. 7. Dispersion of the bare plasmon energy of Zn, corrected for the effects of decreasing core-level oscillator strength. The dashed line gives the dispersion expected from the RPA.

$$\epsilon(\omega)=1-\frac{\omega_p^2}{\omega^2+i\Gamma\omega}+\frac{f_d}{2\omega(\omega+i\gamma)}\times\left[\frac{E_d^2}{\omega(\omega+i\gamma)}\ln\left(\frac{E_d^2}{E_d^2-\omega^2-i\gamma\omega}\right)-1\right]. \quad (3)$$

The first two terms describe the usual Drude expressions for the free charge carriers with a plasma frequency ω_p and a scattering rate Γ.

Figure 6 shows the result of our model calculation for the loss function $\mathrm{Im}[-1/\epsilon(\omega)]$ on the right hand-side, compared to experimental spectra on the left-hand side. For the first model curve (upper panel) we have chosen the set of parameters $\hbar\omega_p=12.7$ eV, $\hbar\Gamma=0.5$ eV, $E_d=9.4$ eV, $f_d=7.5$ eV, and $\hbar\gamma=0.1$ eV in order to achieve a qualitative similarity to the measured spectrum for $q=0.1$ Å^{-1}. It is obvious that such a crude model cannot describe the EELS spectrum in full detail, but the qualitative agreement is evident. Note the low value of the damping parameter $\hbar\Gamma=0.5$ eV. The broadening of the structure in the model function is caused by a complex interplay between the real and imaginary parts of the dielectric function in this energy region. It is not possible to increase the model plasmon width alone without destroying the good qualitative agreement between the experimental and the model curves. But it should be remembered that the value of the experimental plasmon width is somewhat overestimated because the finite resolution of the spectrometer and the background absorption have not been rigorously taken into account. In the lower panel of the simulation, only the value for the core-excitation oscillator strength f_d has been reduced by 50% in order to simulate the behavior of the experimental oscillator strength which has been derived from the sum-rule argument. The other parameters were kept constant. A strong decrease of the core-level feature can be seen as well as a strong shift of the plasmon peak to lower energies. Furthermore, the model plasmon feature has significantly sharpened; the plasmon width is lowered in the model curve by about a factor of 2.5. Besides the shift of the central position and the underestimated linewidth, a qualitative similarity with the corresponding experimental $q=0.6$ Å^{-1} spectrum is found. It should be remarked that the shift of the plasmon position is not only a consequence of the above given model. A simple Drude-Lorentz model, where one Lorentzian with strength f_d describes the core-level excitations, leads to a similar redshift of the plasmon energy. But with such a crude model, not even a qualitative agreement between measured and calculated loss functions can be achieved.

The shift of the plasmon line to negative energies due to the decrease in the oscillator strength of the core-level excitations reduces the usual plasmon dispersion, which in general for metals leads to a quadratic increase in the plasmon position with increasing momentum transfer, i.e., to a positive dispersion. Thus the slightly negative dispersion of the measured plasmon line at low momentum transfer can be explained by a superposition of two effects: a positive dispersion as usual for metals and the shift to lower energies induced by the decrease of core-level oscillator strength. In order to obtain the intrinsic dispersion of the bare plasmon in Zn, we calculated the plasmon position by shifting the measured plasmon energy to higher values, in accordance with the results of our model calculations. Figure 7 shows the resulting dispersion plotted as a function of the squared momentum transfer. A clear positive quadratic dispersion $[\omega_p(q)=\omega_p(0)+(\hbar/m)\alpha q^2]$ is obtained with a dispersion coefficient $\alpha=0.5$, which is slightly higher than the theoretical value $\alpha_{\mathrm{RPA}}=0.42$. The absolute value of the above dispersion must be interpreted with care, because its value may be influenced somewhat by the model's assumptions and the determination of the core-level oscillator strength. However, in general it can be stated that the dispersion parameters for metals having shallow core levels are often higher than the predicted RPA values. The underlying reason is still unclear, but most likely it is related to the presence of the core-levels.

With the same argument for the plasmon dispersion, the decrease of the measured plasmon linewidth (Fig. 3) can be

explained. It decreases by a factor of 1.5 when increasing the momentum transfer from 0.1 to 0.6 $Å^{-1}$. The decrease of the oscillator strength causes a sharpening of the plasmon feature in the model by a factor of 2.5. This means that the intrinsic linewidth independent of the influence of the core-levels would *increase* by a factor of 1.6, a similar value to that obtained in Al.[1] This result supports the considerations expressed above—the plasmon in Zn is quite normal and comparable to that of simple metals, but it is screened by the effects of a drastically momentum-dependent oscillator strength of the core-level excitations.

The most important question remains unanswered: what causes the strong decrease in the oscillator strength of the core-level excitations? The small dispersion in the *d* band which was measured by angular dependent photoemission spectroscopy to be 0.17 eV along the hexagonal axis[13] is too small to account for such a reduction of the combined density of states. Thus the strong decrease must be explained by a strong decrease of the transition matrix elements with increasing momentum transfer. The decrease of the oscillator strength is unusual insofar as it has not been observed[9] in several other metals having shallow core levels such as, for example, Sn and Cd. One interesting point which may probably have a significant influence is the hybridization of the $3d$ bands with the lower part of the conduction band. In the case of post-transition metals this kind of intersection is found only for Zn and Hg. The loss function of Hg is different insofar as the experimental plasmon energy is below the core-level excitation energy. Thus the plasmon in the long-wavelength limit is shifted to significantly lower energies compared to the free-electron value, but it is well pronounced as in the case of other metals with shallow core levels.[23] Unfortunately, no experimental data regarding the plasmon dispersion in Hg and the momentum dependence of the core-level excitations can be found in the literature.

Another case in which *d* bands cross the conduction band can be found in the case of the noble metals. The most intensively investigated member of this family is Ag. The proximity of the free-carrier plasmon and the core-level excitation leads to two plasmonlike features in the loss function[7]—one at 3.8 eV and the other at 8 eV. Both of these features show a strong dispersion, so that the classification in terms of core-level excitation for the lower-energy peak is somewhat problematic. The two structures should be seen more as a coupled system, i.e. as belonging to one type of excitation.[7] Nevertheless we analyzed data for the momentum-dependent loss function of Ag (Ref. 24) in order to investigate the momentum dependence of the spectral weight of the peak at 3.8 eV in Ag, and found no significant changes, in contrast to the behavior found in Zn.

This means that the situation in the case of Zn seems to be quite unique and unusual: a theoretical explanation of the behavior of the core-level oscillator strength is necessary. Probably, calculations of the dielectric properties using an accurate band-structure for Zn could give more insight into this problem.

IV. SUMMARY

We have investigated the momentum-dependent dielectric response of Zn by measuring the loss function of polycrystalline thin films. It is shown that a collective plasma oscillation exists at 14.2 eV in this metal despite the presence of shallow core levels with an excitation energy just below the plasmon energy, which could offer a possible decay channel for the plasmon. The plasmon line is significantly broadened, and thus not as narrow as in other post-transition metals.

With increasing momentum transfer, the oscillator strength of the core-level excitation decreases strongly, which compensates for the plasmon dispersion at low momentum transfer. Additionally, with increasing q, the plasmon feature becomes more pronounced in the spectra. We have shown that upon deconvoluting the effects of changes in the core-level oscillator strength a quite usual behavior of the plasmon characterized by a dispersion coefficient slightly higher than the RPA value and an increase in the linewidth with increasing momentum transfer can be found. The values obtained are similar to those obtained elsewhere from measurements on other post-transition metals.

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