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Transmittance and optical constants of Ho films in the 3–1340 eV spectral range

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The optical constants $n$ and $k$ of holmium (Ho) films were obtained in the 3–1340–eV range from transmittance measurements performed at room temperature. Thin films of Ho with various thicknesses were deposited by evaporation in ultra high vacuum conditions and their transmittance was measured in situ. Ho films were deposited onto thin C-film substrates supported on high transmittance grids. Transmittance measurements were used to obtain the extinction coefficient $k$ of Ho films. The refractive index $n$ of Ho was calculated with Kramers–Kröning analysis; in order to do this, $k$ data were extrapolated both on the high and on the low energy parts of the spectrum by using experimental and calculated $k$ values available in the literature. Ho, similar to other lanthanides, has a low-absorption band below the $O_{2,3}$ edge onset; the lowest absorption was measured at $\sim 22$ eV. Therefore, Ho is a promising material for filters and multilayer coatings in the energy range below the $O_{3,3}$ edge in which most materials have a large absorption. Good consistency of the data resulted from the application of $f$ and inertial sum rules. © 2011 American Institute of Physics. [doi:10.1063/1.3556451]

I. INTRODUCTION

Until recently, lanthanides had not been fully characterized in the extreme ultraviolet (EUV)-soft x-rays. However, an increased interest has grown on these materials with the recent characterization of Yb (Refs. 1 and 2), La (Refs. 3 and 4), Tb (Refs. 3 and 4), Gd (Refs. 5 and 4), Nd (Refs. 4 and 5), Ce (Ref. 6), Pr (Ref. 7), Eu (Ref. 8), Dy (Ref. 4), Tm (Ref. 9), and Lu (Ref. 10), and of materials with close chemical properties such as Sc (Refs. 11–14) and Y (Ref. 15). This paper addresses the optical properties of Ho films in the 3–1340 eV range. The optical properties in this energy range are characterized by the high energy tail of the valence electrons and by the presence of two intense $O_{2,3}$ and $N_{4,5}$ absorption bands, in order of increasing binding energy, due to the excitation of 5p, and 4d electrons, respectively, above the Fermi level.

Scare data are available on the optical properties of Ho in the UV to soft x-rays. Gríbovskii and Zimkina determined the mass absorption coefficient of most rare-earth elements in the 70–500 eV range, which encloses Ho $N_{4,5}$ edge. Vicentin et al. performed transmittance measurements on Ho films and other lanthanides and obtained the absorption coefficient at the $M_{4,5}$ edge. Ott et al. measured the optical constants of a Ho film at the $M_{4,5}$ edge through reflectance measurements performed at 40 K. Zimkina et al. and Fomichev et al. performed absorption measurements and provided data of the product of the absorption coefficient times the film thickness in the 60–460 eV and 161–180 eV ranges, respectively; however, these papers cannot be directly taken for absolute reference since the absorption coefficient cannot be deduced. Pétárik measured the absorption of thin films of Ho in the 1.5–6 eV range and provided data of the product of the absorption coefficient times the film thickness. Sugar calculated the relative positions of the $4d^{10}4f^{14}4I_{15/2}$ to $4d^{10}4f^{13}$ transitions and compared them with the peaks close to $N_{4,5}$ reported in Ref. 19. Fischer and Baun obtained absorption spectra of lanthanides and lanthanide oxides at the $M_{4,5}$ edges; they only plotted the data for the oxides but they stated that the spectrum did not show any difference between metal and oxide; however, no absorption scale was plotted. Thole et al. plotted absorption of lanthanide samples including Ho with focus on a small energy range in the region of the $M_5$ edge aiming at line shape analysis to determine the multiplet components contributing to the absorption peak; since the preparation of the samples is not clearly described and the ordinates in the plotted figures are not clear, the data can only be used qualitatively for the position of the absorption peaks. Tracy...
obtained spectra of vapors of Ho and other lanthanides in the 
~21–40 eV range, and reported relative absorption cross-
section plots. Padalia et al.26 obtained absorption spectra of 
Ho and other lanthanides at L2,3 edges. Materlik et al.27 measured L-edge absorption spectra of Ho and other lantha-
nides. In the low-energy range covered here and at lower 
energies, Weaver and Lynch28 measured the absorptivity of 
oriented single crystals of Ho and other lanthanides in the 
0.2–4.4 eV range at 4.2 K; starting with these data, the com-
pound dielectric constant and the optical constants $n, k$ in the 
0.1–5 eV range were reported in two crystallographic direc-
tions.39 Krizek and Taylor30 provided data of the optical con-
ductivity and $\epsilon_1$ of Ho and other lanthanides obtained from 
elliplism measurements in the 0.35–2.5 eV range at and 
below room temperature. Krizek et al.31 reported Drude pa-
rameters for polycrystalline films of Ho and other lantha-
nides. Weber32 reported infrared data on reflectivity and 
conductivity of single crystals and of thin films of Ho at vari-
ous temperatures.

Other than optical measurements, Bakulin et al.33 mea-
sured the characteristic energy losses of electrons for samples of 
Ho and other lanthanides; they determined the excitation 
energies of the plasma oscillations and the interband excita-
tions. Trebbia and Colliex34 performed electron-energy-loss 
spectroscopy on films of Ho and other lanthanides and they 
reported the oscillator strength close to the N4,5 edge. Colliex 
et al.35 measured the energy loss spectra of electrons trans-
mitted through thin films of Ho and other rare-earth metals 
and their compounds and reported the energies of the plasmon peaks. Borovskii and Komarlov36 obtained the 
absorption coefficient of Ho and other lanthanides from elec-
tron-energy-loss spectra; the data covered the N4,5 range but 
were reported without units. Strasser et al.37 reported 
reflection electron-energy-loss spectra of films of Ho and other 
lanthanides in the region around N4,5 edge. Della 
Valle and Modesti38 reported electron-energy-loss 
spectra of Ho and other lanthanides. Bonnelle et al.39 
reported photoelectron spectra of Ho2O3 in the valence 
region and the 4d region. Kaindl et al.40 obtained the x-ray 
absorption through measurements of total electron yield of 
many compounds including Ho2O3 at M4,5 edge. Sugar et al.41 
performed x-ray photoabsorption spectra of HoF3 and 
other lanthanide fluorides at M4,5 edge from measurements of 
total electron yield. Dzio¨nk et al.42 measured the photon 
yield spectra generated by EUV radiation on atomic 
beams of Ho and other lanthanides. Electron-energy-loss 
spectroscopy in reflection mode of Ho and other lantha-
nides was investigated by Netzter et al.43 Nagao and Igaras-
hi44 calculated the absorption coefficient of Ho at the 
M5 and M6 edges and reported them in arbitrary units. 
Henke et al.45 obtained a semiempirical set of data in the 
30–10 000 eV range (later extended to 30–6000 eV).46 In 
addition to the above references, Weaver et al.29 reviewed 
published data on the optical constants of Ho and other lantha-
nides.

This paper is aimed at providing accurate data on pure 
Ho samples in a broad spectral range in view of the scarce 
and disperse data in the literature. It is organized as follows. 
A brief description of the experimental techniques used in 
this research is given in Sec. II. Section III presents transmitt-
dance data, extinction coefficient of Ho calculated from trans-
mittance, and dispersion obtained using Kramers–Krönig 
(KK) analysis; the consistency of the data gathered in this 
research is also evaluated.

II. EXPERIMENTAL TECHNIQUES

A. Sample preparation

Both Ho film deposition and characterization were per-
formed under ultrahigh vacuum (UHV) at bending magnet 
for emission absorption and reflectivity Bending magnet for 
Emission Absorption and Reflectivity (BEAR) beamline of 
ELETTRA synchrotron (Trieste, Italy).47 Ho films were de-
posited onto 5nm-thick C films supported on 117 mesh Ni 
grids with 88.6% nominal open area (pitch of 216 µm). The 
procedure for C film preparation was reported elsewhere.43 
Ho films were deposited with a TriCon evaporation source,48 
in which a small Ta crucible is bombarded by electrons that 
impinge on the crucible wall. Ho granules of 99.98% purity 
from LTS Chem. Inc. were used. The crucible-sample dis-
tance was 200 mm. Deposition rate was ~4 nm/min. Cham-
ber pressure during deposition was ~2×10−7 Pa. Ho films 
were deposited onto room-temperature substrates. Film 
thickness was monitored with a quartz crystal microbalance 
during deposition. A witness glass substrate was placed close 
to the grid-supported C film to get coated simultaneously 
with a similar Ho film thickness. The distance on samples 
between the area of transmittance measurements and that of 
reflectance measurements was less than 10 mm. Reflectance 
versus the incidence angle was measured on the witness sam-
ple at the energy of 98 eV and the angular positions of the 
minima and maxima were used to calculate the Ho film 
thickness. Since reflectance measurements were performed 
far from absorption edges, Henke optical constants46 could 
be used in this calculation. Henke data were downloaded 
from the website of the Center for X-Ray Optics (CXRO) at 
Lawrence Berkeley National Laboratory.49

B. Experimental setup for transmittance 
measurements

Transmittance measurements were performed at BEAR 
beamline with a vertical exit slit of 100 µm (above 24 eV) 
and 450 µm (below 24 eV); the monochromator spectral 
resolution $E/\Delta E$ varied between 500 and 2000, depending on 
slit widths. The suppression of higher orders was achieved 
using quartz, LiF, In, Sn, Al, and Si filters at specific ranges 
below ~100 eV, and choosing a plane mirror-to-grating 
deviation angle in the monochromator setup that minimized 
the higher-order contribution at energies above 100 eV. The 
beam cross section at the sample was about $0.7 \times 1.5 \text{ mm}^2$ 
FWHM.

The measurements were performed in the BEAR spec-
troscopy chamber;50 a gate valve separates this chamber 
from the preparation chamber, where samples were prepared 
in situ. Two C substrates were used and their transmittance 
was measured previously to Ho deposition. Three and two 
successive Ho coatings of various thicknesses were
accumulated upon the first and the second substrate, respectively, without breaking vacuum. Each sample was transferred back and forth between the deposition chamber and the measurement chamber, always under UHV, for the deposition of the successive Ho layers and their characterization. Transmittance measurements were performed onto samples at room temperature. For each film, uniformity evaluations were performed. We estimate that the overall uncertainty in the transmittance measurements is of the order of 2%. At energies above 18 eV, fluctuations of the photon beam during transmittance measurements were recorded with a 100 V biased, Au mesh. These fluctuations were canceled by normalizing the recorded beam intensity to the mesh current. At energies below 18 eV, fluctuations were canceled by normalization with respect to the ring current.

III. RESULTS AND DISCUSSION

A. Transmittance and extinction coefficient of Ho

We measured the transmittance of Ho films with the following thicknesses: 19.1, 28.7, 39.5, 64.7, and 125.9 nm. The transmittance of the Ho films normalized to the transmittance of the uncoated substrate is plotted in Fig. 1. There are three high-transmission bands peaked at \( \frac{1}{C24} 1330 \) (on the edge of our measurements), \( \frac{1}{C24} 156.5 \), and \( \frac{1}{C24} 21.5–22 \) eV, right below Ho M\(_{5,4} \), N\(_{4,5} \), and O\(_{2,3} \) edges, respectively. The low-energy band of relatively large transmittance extends within \( \frac{1}{C24} 17–23 \) eV. Close large-transmittance bands have been measured for other rare earths; hence Ho, as other lanthanides such as La, Ce, Pr, Nd, Eu, Gd, Tb, Dy, Tm, Yb, and Lu, is a promising material for transmittance filters or multi-layer spacers for the extreme ultraviolet in the \( \frac{1}{C24} 17–23 \) eV spectral range, where there has been a lack of low-absorbing materials until recently. A small oscillation at \( \frac{1}{C24} 315 \) eV can be attributed to Ho N\(_3\) edge. The slight oscillations at \( \sim 100, \sim 285, \sim 405, \sim 460, \) and \( \sim 537 \) eV are related to data normalization, due to the fact that at these energies there is an abrupt decrease of the signal due to the presence of the Si filter, to carbon contamination of the optics, and to the slight presence of N, Ti, and O either at the optics, at the detector, or on the sample.

If the contribution to transmittance coming from multiple reflections inside the Ho film is negligible, the extinction coefficient \( k \) (the imaginary part of the complex refractive index) can be calculated from transmittance with the following equation:

\[
\ln \left( \frac{T_{fs}}{T_s} \right) \approx A - \left( \frac{4\pi k}{\lambda} \right) \cdot d,
\]

where \( T_s \) and \( T_{fs} \) represent the transmittance of the uncoated substrate and of the substrate coated with a Ho film, respectively; \( \lambda \) is the radiation wavelength in vacuum; \( d \) stands for the Ho film thickness. Equation (1) is a straightforward derivation of the wellknown Beer–Lambert law. \( A \) is a constant for each energy and encompasses the terms that involve reflectance, in the assumption that multiple reflections are negligible.

\( k \) of Ho films was calculated by fitting the slope of the logarithm of transmittance versus thickness at each energy using Eq. (1). Examples of transmittance measurements versus the film thickness for five photon energies are given in Fig. 2, along with their fittings. \( k \) data so obtained are represented in Fig. 3 versus the photon energy. Gribovskii data\(^{16} \) and the semiempirical data of Henke\(^{46,49} \) are also plotted in Fig. 3. The aforementioned presence of Si, C, Ti, N, and O oscillations at the Si L\(_{2,3} \), C K, Ti L\(_{2,3} \), N K, and O K edges on transmittance has weakened or disappeared on \( k \) because measurements on samples of different Ho thicknesses with
The density of Ho films is needed to calculate Henke data. The density of thin films may be somewhat lower than the reported data for the bulk material. To do this we deposited a thin film of Ho onto an Al foil. We weighed the Al foil both before and after the Ho deposition with a precision of $\pm 1 \times 10^{-5}$ g. The thickness of the Ho film was measured by Tolansky interferometry on a witness sample. We measured the surface area of the deposit with an optical comparator. We obtained a density of 8.33 ± 0.25 g/cm$^3$ for the Ho film. Several tabulated values for bulk Ho were found, most of them close to 8.80 g/cm$^3$. Hence, the measured density of the film is slightly smaller than that of bulk Ho. The density value measured for the thin film was used to calculate Henke data.

When reflectance is not negligible, the application of Eq. (1) to calculate $k$ through the log of transmittance versus thickness may result in uncertainties. In order to overcome this, we proceeded in an iterative way. For the first iteration, initial $k$ values were obtained using the slope method. These values, along with $k$ data in the rest of the spectrum, were used to obtain the refractive index $n$ (the real part of the complex refractive index) with KK analysis (KK analysis is described in Sec. III B). Once a first set of data $\{n(E), k(E)\}$ was available, the transmittance ratio of the C/Ho bilayer to the single C film was calculated with the usual equations based on Fresnel coefficients. This transmittance ratio was compared with the measured data; the difference between measured and calculated transmittance gave us an estimate to modify $k$. This modified value was a second estimate of $k$, from which a second estimate of $n$ was obtained with KK analysis. This procedure can be iterated until the best match to transmittance data is obtained. The optical constants of the single C film at this same range had been previously calculated with a similar procedure starting with $k$ obtained from the transmittance of an uncoated C substrate. The iterative method was applied in the 3–40 eV range. The $k$ data plotted in Fig. 3 were somewhat modified at 3 eV in an attempt to better match literature data.

In the calculation of $k$ in the range below ~100 eV, transmittance data of the two thickest films was found to be somewhat deviated from the data coming from the three thinnest films. Furthermore, $k$ data calculated with all samples was found to deviate with respect to Henke data in the range between O$_{2,3}$ edge and ~100 eV. Therefore, in the calculation of $k$ we decided to use only the three thinnest films below 100 eV, whereas all five films were used at N$_{4,5}$ edge and above, with a smooth connection in between; this resulted in a better match with Henke data below 100 eV.

$k$ values at the O$_{2,3}$ edge and around are presented in Fig. 4. The smallest value of $k$ is obtained at ~22.0 eV. This minimum is close to the ones obtained for other rare earths: Ce (Ref. 6) at 16.1 eV, La (Ref. 3) at 16.5 eV, Eu (Ref. 8) at 16.7 eV, Pr (Ref. 7) at 16.87 eV, Nd (Ref. 5) at ~17 eV, Tb (Ref. 3) at ~19.5 eV, Gd (Ref. 5) at ~19.7 eV, Dy (Ref. 4) at ~20.2 eV, Yb (Refs. 1 and 2) at 21.2 eV, Tm (Ref. 9) at 23 eV, Lu (Ref. 10) at 25.1 eV, and Sc (Ref. 11) (neighbor in the periodic table) at 27 eV. As with other lanthanides, optical properties of Ho in this range are promising for its use in transmittance filters or reflective multilayers. However, Ho, as the other lanthanides, is a reactive material, and this may result in the need to develop a protective layer.

Figure 5 displays $k$ around Ho N$_{4,5}$ edge, along with experimental data of Gribovskii and Zimkina and semiempirical data of Henke et al. The current data show a structure of three narrow peaks at 157.75, 158.88, and 160.88 eV, and two

FIG. 3. (Color online) Log-log plot of the extinction coefficient of Ho as a function of photon energy, along with the data of Gribovskii (Ref. 16) and the data of Henke et al. (Ref. 49).

FIG. 4. (Color online) The extinction coefficient of Ho as a function of photon energy at the small energy range, along with the data of Henke et al. (Ref. 49).
broader and higher peaks at 166.3 and 171.0 eV. The peaks are related to transitions from 4d to 4f shells. Fomichev et al.\textsuperscript{20} reported three peaks at 155.8, 156.9, and 158.8 eV, and their data does not reach the energy range of our broader peaks. Sugar\textsuperscript{22} calculated the position of the peaks. In addition to the peaks measured by Fomichev et al.,\textsuperscript{20} Sugar obtained 9 peaks between 161.7 and 174.6 eV that he related to a single experimental peak observed by Zimkina et al.\textsuperscript{19} at $\sim$167 eV. The latter can be associated with our two broader peaks. Gribovskii’s data match well our data, but the former data have a much coarser sampling. Hence, regardless of the precision of the exact peak energies, we provide here first quantitative $k$ data at both narrow and broad peaks at the N$_{4,5}$ range.

At $\sim$1000 eV (see Fig. 1), two transmittance-versus-energy curves intersect for reasons that are not well understood. In the calculation of $k$ at these high energies we decided to use the measurements on all samples since we had no guide to reject any data.

### B. Refractive index calculation through dispersion relations

The refractive index $n$ of Ho was calculated using KK dispersion relations:

$$n(E) - 1 = \frac{2}{\pi} P \int_0^\infty \frac{E'k(E')}{E'^2 - E^2} dE',$$

where $P$ stands for the Cauchy principal value. The application of Eq. (2) to calculate $n$ requires the availability of $k$ data over the whole spectrum, so that we extended the present data with the available data in the literature and extrapolations.

At the Ho M$_{4,5}$ edge we could use the data of Vicentin et al.\textsuperscript{17} and Ott et al.\textsuperscript{18} $k$ data at the M$_{4,5}$ edge can be immediately obtained from the absorption coefficient reported by Vicentin et al.\textsuperscript{17} Ott et al.\textsuperscript{18} reported both optical constants at the M$_5$ edge. However, Vicentin’s M$_5$ peak was about twice the value of that of Ott et al. In principle, the data published by Vicentin et al. were obtained in excellent conditions to result in precise data. Since Vicentin’s paper reported data not only of Ho but also Gd, Dy, and Er, we could compare their experimental results to literature data. In a separate paper devoted to Er optical constants,\textsuperscript{51} we obtained that Vicentin’s $k$ value at Er M$_5$ edge was much larger than our data. Furthermore, Vicentin’s $k$ data at Gd M$_5$ edge was 0.0114, whereas we derived, using the transmittance data reported in Fig. 2 of the paper of Peters et al.,\textsuperscript{52} a value of 0.0074 at this same Gd M$_5$ edge. Hence we suspect that all Vicentin’s data may be somewhat too large. Furthermore, we represented M$_{4,5}$-edge $k$ data of several lanthanides that we have been gathering in this long-run research and we found that Vicentin’s data for Ho was far above the trend of lanthanides; even Ott’s data was somewhat larger than this trend. All the above convinced us not to use Vicentin’s data directly, although we did it indirectly in the following way. We could have used Ott’s data, but they did not report on the M$_4$ edge. Then we merged the two data sets: we used Vicentin’s data but we scaled both their M$_4$ and M$_5$ peaks down in a factor given by the Ott-to-Vicentin’s M$_5$ peak $k$ data ratio. Above the M$_4$ edge we smoothly connected these data with those of Henke. Figure 6 displays the data so constructed, which is referred to as rescaled Vicentin, along with the experimental data of Vicentin et al.,\textsuperscript{17} Ott et al.,\textsuperscript{18} and the semiempirical data of Henke. The inset compares in logarithmic scale the data of Ott et al.\textsuperscript{18} with the original data of Vicentin et al.\textsuperscript{17}

Further extrapolations were as follows. Between 1400 and 3-$10^4$ eV we used Henke data from CXRO’s web.\textsuperscript{46,49} For even larger energies, the calculations of Chantler et al.\textsuperscript{53} were used up to 4.3-$10^5$ eV. The extrapolation to infinity was
performed by keeping constant the slope of the log-log plot of $k(E)$ of Chantler’s data. At energies smaller than the present ones, we used the data of Krizek and Taylor,\textsuperscript{30} from whose conductivity and $\epsilon_1$ data we could immediately obtain $k$ in the 0.38–2.6 eV range. This was preferred over using the data of Weaver and Lynch\textsuperscript{28} because the latter was measured on single crystals, compared to our films, and their use would require an average over the two sets of optical constants measured at the two main axes. The extrapolation to zero energy was performed by fitting a Drude model on Krizek’s data.

Figure 7 displays $k$ data of Ho obtained in the present research along with literature data, calculations, and extrapolations that were gathered for KK analysis.

Figure 8 displays $\delta = 1 - n$ calculated with Eq. (2) using the data plotted in Fig. 7; $n$ and $\delta$ at $O_{2,3}$, and $N_{4,5}$ edges are shown in Figs. 9 and 10, respectively. We also plot $\delta$ that was calculated at the $M_{4,5}$ edge, which is given in Fig. 11. Ott’s data and the semiempirical data of Henke are also plotted for comparison. $\delta$ data obtained at the $M_5$ edge are relatively close to Ott’s data, with a shift in energy of 3.4 eV, which is similar to the energy difference between Vicentin’s and Ott’s data for $M_5$ peak $k$ data.
Planck’s constant. The obtained with the inertial sum rule:

\[ \zeta = \frac{\int_0^{\infty} [n(E) - 1]dE}{\int_0^{\infty} n(E) - 1|dE}. \tag{5} \]

Shiles et al.\(^5^4\) suggested that a good value of \( \zeta \) should stand within \( \pm 0.005 \). An evaluation parameter \( \zeta = -4 \times 10^{-4} \) was obtained here with the \( n \) data calculated in this research. Therefore, the inertial sum test is well within the above top value, which, along with the result obtained above for the \( f \) sum rule, suggest good consistency of \( n \) and \( k \) data.\(^5^7\)

IV. CONCLUSIONS

The transmittance of thin films of Ho deposited by evaporation has been measured \textit{in situ} in the 3–1340 eV photon energy range under UHV conditions. The extinction coefficient \( k \) of Ho has been calculated from transmittance measurements in the same spectral range. Ho features an absorption minimum at 22 eV. This relatively low absorption at this spectral range makes Ho a promising candidate for transmittance filters and reflective multilayers. Given the reactivity of Ho, as with other lanthanides, a surface passivation method is expected to be required to prevent surface instability of Ho in contact with atmosphere.

The refractive index \( n \) of Ho in the same range was obtained with KK analysis over an extended spectral range.

The current data encompass the extinction coefficient and the refractive index data of Ho at the N\(_{4,5} \) and O\(_{2,3} \) edges. It is also proposed a rescaling for the data available in the literature at the M\(_{4,5} \) edge.

The evaluation of \( f \) and inertial sum rules shows good consistency of the optical constants of Ho.

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