Transmittance and optical constants of erbium films in the 3.25 – 1580 eV spectral range

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The optical constants of erbium (Er) films were obtained in the 3.25–1580 eV range from transmittance measurements performed at room temperature. Thin films of Er were deposited by evaporation in ultra high vacuum conditions and their transmittance was measured in situ. Substrates consisted of a thin C film supported on a grid. Transmittance measurements were used to obtain the extinction coefficient k of the Er films. The refractive index n of Er was calculated using the Kramers–Krönig analysis. k data were extrapolated both on the high- and low-energy parts of the spectrum by using experimental data and calculated k values available in the literature. Er, similar to other lanthanides, has a low-absorption band below the O₂,₃ edge onset; the smallest absorption was measured at ∼22.5 eV. Therefore, Er is a promising material for filters and multilayer coatings in the energy range below the O₂,₃ edge, in which materials typically have an absorption stronger than at other energies. Good consistency of the data resulted from the application of f and inertial sum rules. © 2011 Optical Society of America

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1. Introduction

Until recently, lanthanides had not been fully characterized in the extreme ultraviolet (EUV) and soft x rays. However, an increased interest has grown on these materials with the recent characterization of Yb [1,2], La [3,4], Tb [3,4], Gd [5,4], Nd [5,4], Ce [6], Pr [7], Eu [8], Dy [4], Tm [9], and Lu [10], and of materials with close chemical properties such as Sc [11–14] and Y [15]. This paper addresses the optical properties of erbium (Er) films in the 3.25–1580 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 three intense absorption bands, O₂,₃, N₄,₅, and M₄,₅ in order of increasing binding energy, due to the
excitation of $5p$, $4d$, and $3d$ electrons, respectively, above the Fermi level. Only partial data are available on the optical properties of Er in the UV to soft X-rays. Gribovskii and Zimkina [16] determined the mass absorption coefficient of most rare-earth elements in the 70–500 eV range, which encloses the Er $N_{4.5}$ edge. Vicentin et al. [17] performed transmittance measurements on Er films and other lanthanides in the 1380–1480 eV range and obtained the absorption coefficient in this range, which enclosed the M$_{4,5}$ edges. Zimkina et al. [18] and Fomichev et al. [19] 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. Sugar [20] calculated the relative positions of the 4d$^1$ $4f^{14}$ $4f^{15}$,2 transitions and compared them with the peaks close to N$_{4.5}$ reported in [18], in which the highest peak was found at 174.9 eV. Fischer and Baun [21] obtained absorption spectra of lanthanides and lanthanide oxides at the M$_{5}$ and M$_{4}$ edges, which they found at 1401.7 and 1444.9 eV, respectively, for Er; 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. [22] plotted the absorption of lanthanide samples including Er in the 1395–1455 eV range. Thole’s research aimed at line shape analysis to determine the multiplet components contributing to the absorption peak. Since the preparation of the samples is not precisely described and the ordinates in the plotted figures are not clear, the data can only be used qualitatively for the position of the M$_{4,5}$ absorption peaks, which were found at 1446.3 and 1404.9 eV. Tracy [23] obtained spectra of vapors of Er and other lanthanides in the ~21–40 eV range, and reported relative absorption cross-section plots. Padalia et al. [24] obtained absorption spectra of Er and other lanthanides at the L$_{2,3}$ edges, which are at energies larger than the current range (8361.2, 9268.4 eV); Materlik et al. [25] also measured L-edge absorption spectra of Er and other lanthanides. In the low-energy range covered here and at lower energies, Weaver and Lynch [26] measured the absorptivity of oriented single crystals of Er and other lanthanides in the 0.2–4.4 eV range at 4.2 K. Starting with this data, the complex dielectric constant and the optical constants $n$, $k$ in the 0.1–5 eV range were obtained in two crystallographic directions [27]. Krizek and Taylor [28] provided data of the optical conductivity of Er and other lanthanides obtained from ellipsometry measurements in the 0.35–2.5 eV range. Knyazev and Noskov [29] obtained the optical constants $n$ and $k$ of Er films in the 0.06–4.4 eV range, both at room and at liquid nitrogen temperatures, from optical measurements using a polarimetric method; they used bulk polycrystalline samples that had been polished. Jiles and Staines [30] measured the piezoreflectance of the thin films of Er and other lanthanides. The authors also plotted the reflectance of Er in the ~0.7–7 eV range that they attribute to a previous literature paper (present [29]), but we did not find such reflectance in the referenced paper or elsewhere. The authors performed a Kramer–Kronig (KK) analysis in the referred range [30] and plotted data of the imaginary part of the dielectric constant $\varepsilon_2$. Knyazev and Bolotin [31] characterized Er and Tm single crystals by ellipsometry both at 78 and 293 K, from which they calculated Er optical constants in two crystallographic directions in the 0.2–5.6 eV spectral range. Öncan et al. [32] measured the dependence of the effective dielectric constant $\varepsilon_2$ on the thickness of thin films of Er in the 1.55–5.6 eV range.

Other than optical measurements, Bakulin et al. [33] measured the characteristic energy losses of electrons for samples of Er and other lanthanides for energies up to 50 eV; they determined the excitation energies of the plasma oscillations and the interband excitations. Trebbia and Colliex [34] performed electron energy loss spectroscopy on films of Er and other lanthanides, and they reported the oscillator strength close to the N$_{4.5}$ edge. Colliex et al. [35] measured the energy loss spectra of electrons transmitted through thin films of Er and other rare-earth metals and their compounds up to ~50 eV, and reported the energies of the plasmon peaks. Bertel et al. [36] took electron energy loss spectra of Er films and bulk up to 60 eV; they reported the loss peaks for pure Er and for Er exposed to doses of O$_2$ and H$_2$. Strasser et al. [37] reported reflection electron energy loss spectra of films of Er and other lanthanides in the region around the N$_{4.5}$ edge. Della Valle and Modesti [38] reported reflection electron energy loss spectra of Er and other lanthanides up to energies of 15 eV for various primary electron energies; the spectra were characterized by sharp peaks due to exchange-excited dipole-forbidden $f$–$f$ transitions. Bonnelle et al. [39] reported photoelectron spectra of Er$_2$O$_3$ in the valence region for energies below 40 eV and in the 4d region (170–200 eV). Kaindl et al. [40] obtained x-ray absorption through measurements of the total electron yield of many compounds including Er$_2$O$_3$ at M$_{4.5}$ edge (1390–1450 eV). Sugar et al. [41] performed x-ray photoabsorption spectra of ErF$_3$ and other lanthanide fluorides at the Er M$_5$ and M$_4$ edges (1392–1412, 1432–1447 eV) from measurements of the total electron yield. Dziokn et al. [42] measured the photon yield spectra generated by EUV radiation on atomic beams of Er and other lanthanides in the 24–36 eV range. Electron energy loss spectroscopy in the reflection mode of Er and other lanthanides was investigated by Netzer et al. [43] in the energy range up to 45 eV. Broden [44] performed photoemission measurements on Er in the photon energy range of 4 to 21 eV. Henke et al. [45] obtained a semimembrary set of data in the 30–10,000 eV range (later extended to 30,000 eV [46]). In addition to the above references, Weaver and Lynch [26] and Ward [47] reviewed published...
data on the optical constants of Er and other lanthanides.

The above review of the available data on the optical constants of Er shows that there are spectral intervals with no data, and there is a lack of a consistent set of data covering at least the EUV. This paper is aimed at providing a consistent set of optical constant data on pure Er samples in the 3.25–1580 eV spectral range. It is organized as follows. A brief description of the experimental techniques used in this research is presented in Section 2. Section 3 presents transmittance data, extinction coefficient of Er calculated from transmittance, and dispersion obtained using KK analysis; the consistency of the data gathered in this research is also evaluated.

2. Experimental Techniques

A. Sample Preparation

Both Er film deposition and characterization were performed under ultra high vacuum (UHV) at the bending magnet for emission absorption and reflectivity (BEAR) beamline of the ELETTRA synchrotron (Trieste, Italy) [48]. Er films were deposited onto 5 nm 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 [13]. Er films were deposited with a TriCon evaporation source [49], in which a small Ta crucible is bombarded by electrons that impinge on the crucible wall. Er lumps of 99.95% purity from LTS Chem, Inc. were used. Pressure during evaporation was less than 9 × 10⁻⁷ Pa. The crucible sample distance was 200 mm. Deposition rate was ~4 nm/min. Film thickness was monitored with a quartz crystal microbalance during deposition. Er films were deposited onto room-temperature substrates. Witness glass substrates for reflectance measurements were placed close to the grid-supported C film and were coated with a similar Er film thickness. The distance on the surface sample between the area of transmittance measurements and that of reflectance measurements was ~10 mm. Reflectance versus the incidence angle was measured on the witness samples at an energy of 100 or 200 eV, and the angular positions of the minima and maxima were used to calculate the Er film thickness. Henke’s optical constants [46] were used in this calculation. Henke’s data were downloaded from the website of the Center for X-Ray Optics (CXRO) at Lawrence Berkeley National Laboratory [50].

B. Experimental Setup for Transmittance Measurements

Transmittance measurements were performed at the BEAR beamline with vertical exit slits of 100 μm (above 24 eV) and 450 μm (below 24 eV); the monochromator spectral resolution E/ΔE varied between ~500 and 2000, depending on the 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 [51]. The beam cross section at the sample, after defocusing to reduce the radiation density on the sample, was about 0.7 mm × 1.5 mm FWHM.

The measurements were performed in the BEAR spectroscopy chamber [52]; a gate valve separates this chamber from the preparation chamber, where samples were prepared in situ. Three C substrates were used, and their transmittance was measured. Three successive Er coatings of various thicknesses were deposited upon the first substrate without breaking vacuum; the other two substrates received only the deposition of a single Er thickness. All transfers from the deposition chamber to the measurement chamber and vice versa were performed under UHV to avoid contamination from atmospheric species. Transmittance measurements were performed on 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 15 eV, fluctuations of the photon beam during transmittance measurements were recorded with a 100 V biased Au mesh. These fluctuations were cancelled by normalizing the recorded beam intensity to the mesh current. Below 15 eV, the normalization to minimize fluctuations was performed with respect to the storage ring current.

3. Results and Discussion

A. Transmittance and Extinction Coefficient of Er

We measured the transmittance of Er films with the following thicknesses: 15.5, 25, 37.5, 52, and 94 nm; the latter film was measured only at some energy ranges due to the limited allocated time. The transmittance of the Er films normalized to the transmittance of the uncoated substrate is plotted in Fig. 1.

![Fig. 1. (Color online) Transmittance of Er films with various thicknesses (in nm) normalized to the transmittance of the uncoated substrate versus the logarithm of photon energy.](image-url)
There are three high transmission bands peaked at \( \sim 1336-1390, 163.5, \) and \( \sim 22-22.5 \) eV, right below the Er M\(_5\), N\(_{4.5}\), and O\(_{2.3}\) edges, respectively. The low-energy band of relatively large transmittance extends within \( \sim 19-23.5 \) eV, close to transmittance bands measured in other rare earths. Hence, Er, 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 multilayer spacers for the extreme ultraviolet in the \( \sim 19-23.5 \) eV spectral range, where there has been a lack of low-absorbing materials until recently. A small feature at \( \sim 285, \sim 460, \) and \( \sim 535 \) eV are related to data normalization due to traces of carbon contamination of the optics, and to the slight presence of Ti, and O either on the detector or on the sample, respectively. The presence of Ti originates in the titanium sublimation pump.

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

\[
\ln \left( \frac{T_f}{T_s} \right) \approx A - \left( \frac{4nk}{\lambda} \right) \cdot d, \tag{1}
\]

where \( T_s \) and \( T_f \) represent the transmittance of the uncoated substrate and of the substrate coated with an Er film, respectively; \( \lambda \) is the radiation wavelength in vacuum; \( d \) stands for the Er film thickness. Equation (1) is a straightforward derivation of the well-known 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 the Er films was calculated by fitting the slope of the logarithm of transmittance versus thickness at each energy using Eq. (1); the data are represented in Fig. 2 [53]. The semiempirical data of Henke et al. [46, 50], also plotted in Fig. 2, were calculated assuming the bulk density of Er, i.e., \( 9.066 \) g/cm\(^3\). \( k \) data of Gribovskii et al. [16] and of Knyazev et al. [29] are the only data found in the literature within the plotted spectral range, and they are also displayed. The aforementioned presence of C, Ti, and O oscillations at the C K, Ti L\(_{3,2}\), and O K edges is less significant on \( k \) than on transmittance, because samples of different Er thicknesses with a similar presence of contaminants (either on the sample, on the detector, or in the light path) or with artifacts coming from normalization at transmittance calculation will tend to cancel out in the calculation of \( k \) with the slope method.

In the above calculation, \( k \) data obtained with the film thicknesses calculated from reflectance data resulted in values smaller than Henke’s \( k \) data in the whole range, which also resulted in too low a consistency parameter that is defined in Subsection 3.C. Hence, we concluded that real film thicknesses should be somewhat smaller than the ones we had used. We obtained new film thickness data in the following way. The transmittance-versus-energy curve for each thickness above \( 200 \) eV was fitted to the data calculated with Henke’s optical constants by varying the film thickness until the best match was obtained.

The new thicknesses were used in the calculation of \( k \) with our transmittance data, which is plotted in Figs. 2 and above. Film thicknesses plotted in Fig. 1 are those modified to match Henke’s data. The need to modify the film thicknesses that were obtained from reflectance measurements might have been due in part to the difficulties in deriving the correct thickness from the reflectance measurements, and possibly in part to a slightly lower density of our films compared to bulk Er.

When reflectance is not negligible, the application of Eq. (1) to calculate \( k \) through the slope of the logarithm 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 the \( k \) data in the rest of the spectrum, were used to obtain the refractive index \( n \) (the real part of the complex refractive index) with the KK analysis (KK analysis is described in Subsection 3.B). Once a first set of data \( \{n(E), k(E)\} \) was available, the transmittance ratio of the C/Er 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 the 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 3.25–40 eV range. 

\( k \) at the O\(_{2,3}\) edge and below are presented in Fig. 3, along with the data from Knyazev and Noskov \[29\] and the semiempirical data of Henke et al. \[46,50\]. The smallest value of \( k \) is obtained at \( \sim 22.5 \) eV. This minimum is close to the ones obtained for other rare earths: Ce \[6\] at 16.1 eV, La \[3\] at 16.5 eV, Eu \[8\] at 16.7 eV, Pr \[7\] at 16.87 eV, Nd \[5\] at \( \sim 17 \) eV, Tb \[3\] at \( \sim 19.5 \) eV, Gd \[5\] at \( \sim 19.7 \) eV, Dy \[4\] at \( \sim 20.2 \) eV, Yb \[1,2\] at 21.2 eV, Tm \[9\] at 23 eV, Lu \[10\] at 25.1 eV, and Sc \[11\] at 27 eV. As with other lanthanides, optical properties of Er in this range are promising for its use in transmittance filters or reflective multilayers. However, Er, as its neighbors in the periodic table, is a reactive material, and this may result in the need to develop a protective layer.

Figure 4 displays \( k \) around the Er N\(_{4,5}\) edge, along with experimental data from Gribovskii and Zimkina \[16\] and the semiempirical data from Henke \[50\]. The present data show a structure of two narrow peaks at 164.4 and 166.4 eV, and two broader and higher peaks at 168.3, 175.9 eV, with a smaller peak in between at 172.5 eV. The peaks are related to transitions from 4\( d \) to 4\( f \) shells. Fomichev et al. \[19\] reported the same number of peaks at 163.4, 165.1, 167.2, and 174.8 eV (Fomichev's data, normalized to match Gribovskii's data, are also plotted in Fig. 4); a shoulder between the latter two peaks may correspond to the peak we found at 172.5 eV. Our peaks are at an average energy of 1.1 eV larger than Fomichev's ones.

\( k \) at the M\(_{4,5}\) edge is presented in Fig. 5, along with the experimental data from Vicentin et al. \[17\], and Thole et al. \[22\], and with the semiempirical data of Henke \[50\]. Thole's data, not displaying any ordinate units, have been scaled to match the present peak heights; in fact, Thole's data refer to absorption, which is plotted to compare peak shape (although one has to keep in mind that the present figure refers to extinction coefficient) and position. The peak position varies among the different data, our peak positions being relatively centered between the data from Vicentin and Thole. The peak structure in the present data is similar to Thole and Vicentin, in which a small peak at larger energy than the two main peaks in the first two sets of data appears as a shoulder in Vicentin's data.

An important difference between Vicentin and the present data is the much higher double-peak of the former (a factor of 3.5). In principle, the data...
published by Vicentin et al. [17] were obtained in excellent conditions to result in precise data. Since Vicentin’s paper reported data not only of Er but also of Gd, Dy, and Ho, we could compare their experimental results to literature data. In a separate paper devoted to Ho optical constants [54], we discussed that Vicentin’s $k$ value at the Ho M$_5$ edge was much larger than the one of Ott et al. [55], which is discussed in [54]. Regarding Gd, Vicentin’s $k$ data were 0.0114 at the M$_5$ peak, whereas we derived, using the transmittance data reported in the Fig. 2 from Peters et al. [56], a value of 0.0074 at the same Gd M$_5$ edge. Hence, we suspect that Vicentin’s data may be somewhat too large in general. Furthermore, we represented the 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 Er was far above the trend of lanthanides, whereas our $k$ at the main peak is within the general trend of lanthanides.

B. Refractive Index Calculation through Dispersion Relations

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

$$n(E) - 1 = \frac{2P}{\pi} \int_{0}^{E_0} \frac{E'k(E')}{E'^2 - E^2} dE'$$

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. Between 1580 and $3 \cdot 10^3$ eV, we used Henke’s data from the CXRO’s website [50]; the two sets of data were coupled with a smooth connection. For even larger energies, the calculations of Chantler et al. [57] were used up to $4.3 \cdot 10^5$ eV. The extrapolation to infinity was performed by keeping the slope of the log-log plot of $k(E)$ of Chantler’s data constant.

At energies smaller than the present ones, we used the data of Knyazev and Noskov [29]; we selected these data because they covered a wide spectral range, and they measured optical constants for Er films. We coupled Knyazev’s data with the present data with a smooth connection. The extrapolation to zero energy was performed by fitting a Drude model on the Knyazev data.

Figure 6 displays $k$ data of Er that were gathered for KK analysis. Figure 7 [53] displays $\delta = 1 - n$ calculated with Eq. (2) using data plotted in Fig. 6; $n$ or $\delta$ at the O$_{2,3}$, N$_{4,5}$, and M$_{4,5}$ edges are shown in Figs. 8–10, respectively. Only the Knyazev and Henke data are available for comparison.

C. Consistency of Optical Constants

The $f$ sum rule relates the number density of electrons to $k$ (or to other functions); it provides a guidance to evaluate the accuracy of the $k$ data. It is useful to define the effective number of electrons per atom $n_{\text{eff}}(E)$ contributing to $k$ up to given energy $E$:

$$n_{\text{eff}}(E) = \frac{4e_0m}{\pi N_{\text{at}}^2e^2h^2} \int_{0}^{E} E'k(E')dE'$$

where $N_{\text{at}}$ is the atom density, $e$ is the electron charge, $\varepsilon_0$ is the permittivity of vacuum, $m$ is the electron mass, and $h$ is Planck’s constant [58]. The $f$ sum rule expresses that the high-energy limit of the effective number of electrons must reach $Z = 68$, i.e., the atomic number of Er. When the relativistic correction on scattering factors is taken into account, the high-energy limit of Eq. (3) is somewhat modified. The following modified $Z$ was adopted here: $Z' = 66.84$ [57]. The high-energy limit that we obtained by integrating the dataset plotted in Fig. 6 using Eq. (3) was 66.1, which is 1.1% smaller than the above $Z'$ value, which is considered a good match. The main contribution to
$n_{\text{eff}}$ was found to come from the $\sim 1$ to $5 \times 10^5$ eV range. The small difference with $Z/C^3$ may be attributed to inaccuracies in the film thickness determination, in the transmittance measurements, and in the $k$ data used in the energy extrapolations.

A useful test to evaluate the accuracy of the KK analysis is obtained with the inertial sum rule

$$Z_0 \left[ \frac{n(E)}{C^{138}} - \frac{1}{C^{138}} \right] dE = 0, \quad (4)$$

which expresses that the average of the refractive index throughout the spectrum is unity. The following parameter is defined to evaluate how close to zero the integral of Eq. (4) [58] is:

$$\zeta = \frac{\int_0^\infty |n(E) - 1| dE}{\int_0^\infty n(E) - 1| dE}. \quad (5)$$

Shiles et al. [58] suggested that a good value of $\zeta$ should stand within $\pm 0.005$. An evaluation parameter $\zeta = 0.0027$ was obtained here with the dataset plotted in Fig. 6. Therefore, the inertial sum rule test is within the above top value, which along with the result obtained above for the $f$ sum rule, suggests good consistency of the $n$ and $k$ data. A new paper has been recently published with optical constants of Er films [59].

4. Conclusions

The transmittance of the thin films of Er deposited by evaporation has been measured in situ in the 3.25–1580 eV photon energy range under UHV conditions. The extinction coefficient $k$ of Er has been calculated from transmittance measurements in the same spectral range. Er features an absorption minimum at $\sim 22.5$ eV. This relatively low absorption at this spectral range makes Er a promising candidate for transmittance filters and reflective multilayers. Given the reactivity of lanthanides, a surface passivation method may be necessary to prevent surface instability of Er in contact with the atmosphere.

The refractive index $n$ of Er in the same range was obtained with KK analysis over an extended spectral range.

The current $n$ and $k$ data encompass Er M$_{4,5}$, N$_{4,5}$ and O$_{2,3}$ edges.

The evaluation of $f$ and inertial sum rules shows good consistency of the optical constants of Er.

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50. URL: http://www-csro.lbl.gov/optical_constants/.


53. The data are available on request at the following e-mail address: larruquert@io.cfm.ac.cisic.es.


