

Transmittance and optical constants of Lu films in the 3-1,800 eV spectral range

Sergio García-Cortés, Luis Rodríguez-de Marcos, Juan I. Larruquert*, José A. Aznárez, José

A. Méndez

GOLD-Instituto de Física Aplicada-Consejo Superior de Investigaciones Científicas

Serrano 144, 28006 Madrid, Spain

Luca Poletto, Fabio Frassetto

Institute of Photonics and Nanotechnologies-National Council for Research,

via Trasea 7, 35131 Padova, Italy

A. Marco Malvezzi

Dipartimento di Elettronica, Università di Pavia and CNISM, Via Ferrata, 1, I27100 Pavia, Italy

Angelo Giglia, Nicola Mahne

Istituto Officina dei Materiali IOM-CNR Laboratorio TASC, Area Science Park Basovizza, S.S. 14

Km 163.5, 34149 Trieste, Italy

Stefano Nannarone

Dipartimento di Ingegneria dei Materiali e dell Ambiente, Università di Modena e Reggio Emilia,

Via Vignolese 905, I-41100 Modena, Italy

ABSTRACT

The optical constants n and k of lutetium (Lu) films were obtained in the 3-1,800-eV range from transmittance measurements performed at room temperature. These are the first experimental optical constant data of Lu in the whole range. Thin films of Lu with various thicknesses were deposited by evaporation in ultra high vacuum conditions and their transmittance was measured in situ. Lu films

* Author to whom correspondence should be addressed. Electronic mail: larruquert@ifa.cetef.csic.es; fax number: 34 914117651

were deposited onto grids coated with a thin, C support film. Transmittance measurements were used to obtain the extinction coefficient k of Lu films. The refractive index n of Lu was calculated with Kramers-Krönig analysis. k data were extrapolated both on the high and on the low energy sides by using experimental and calculated k values available in the literature. Lu, similar to other lanthanides, has a low-absorption band below the O_{2,3} edge onset; the lowest absorption was measured at ~25.1 eV. Therefore, Lu is a promising material for filters and multilayer coatings in the energy range below the O_{2,3} edge in which most materials have a large absorption. Good consistency of the data was obtained through f and inertial sum rules.

Keywords: Extreme Ultraviolet; Far Ultraviolet; Optical constants; Absorption filters; Multilayers; Optical properties of thin films

1. 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^{1,2}, La^{3,4}, Tb^{3,4}, Gd^{5,4}, Nd^{5,4}, Ce⁶, Pr⁷, Eu⁸, Dy⁴, and Tm⁹, and of materials with close chemical properties such as Sc^{10,11,12,13} and Y¹⁴. This paper addresses the optical properties of Lu films in the 3-1,800-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 M_{4,5} absorption bands, in order of increasing binding energy, and of a weak N_{4,5} band, due to the excitation of 5p, 3d, and 4d electrons, respectively, above the Fermi level.

Almost no previous data on the optical properties of Lu in the UV to soft X-rays has been found for this research. Zimkina *et al.*¹⁵ performed absorption measurements on Lu thin films and provided data of the product of the absorption coefficient times the film thickness in arbitrary units in the ~60-480 eV range. Unfortunately, this paper cannot be directly taken for absolute reference since the absorption coefficient cannot be deduced. Fischer and Baun¹⁶ obtained M_α and M_β emission spectra of lanthanides and lanthanide oxides, including Lu; they only plotted the data for the oxide but they stated that the spectrum did not show any change between metal and oxide. For other lanthanides they also displayed absorption spectra, which were relatively similar to the emission spectra; however, this comparison was not given for Lu. At higher energies than the present research, Padalia *et al.*¹⁷ obtained absorption spectra of Lu and other lanthanides at L_{2,3} edges.

Other than optical data, Bonnelle *et al.*¹⁸ reported photoelectron spectra of Lu₂O₃ in the valence region and the 4d region. Lang *et al.*¹⁹ reported core-level binding energies of Lu measured by x-ray photoelectron spectroscopy. Colliex *et al.*²⁰ measured the energy loss spectra of electrons transmitted

through thin films of Lu and other rare-earth metals and their compounds. For Lu, the authors obtained a main energy-loss peak at 14.9 eV, which they assigned to collective excitations of the upper electron band, and a less intense peak at 41.0 eV, which they assigned to the excitation of the 5p electrons of the metal atom. Onsgaard *et al.*²¹ measured the reflection energy-loss spectra of the (001) surface of a Lu single crystal, and they provided the energies of single electron excitation of Lu. Netzer *et al.*²² presented the electron energy-loss spectra in reflection mode of Lu and the other lanthanides. Manoubi *et al.*²³ determined cross section ratios of rare-earth M_{4,5} to oxygen K for rare-earth oxides including Lu. Henke *et al.*²⁴ obtained a semi-empirical set of data in the 30-10,000-eV range (later extended to 30,000 eV²⁵). Henke compilations could only use the data in arbitrary units of Zimkina *et al.*¹⁵. Due to the lack of data on Lu, Henke *et al.* had to use data on Yb at M_{3,4,5} edges, coming from Combley *et al.*²⁶.

The 5p to nd resonances and the direct 5p photoionization of atomic Lu has been investigated by photoelectron spectroscopy and photoion spectroscopy in combination with monochromatized synchrotron radiation in the vacuum ultraviolet energy region^{27,28}.

In view of the full lack of data, this paper is aimed at providing accurate optical data on pure Lu films in a broad 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 Lu calculated from transmittance, and dispersion obtained using KK analysis; the consistency of the data gathered in this research is also evaluated.

2. Experimental techniques

2.1 SAMPLE PREPARATION

Both Lu film deposition and characterization were performed under ultra high vacuum (UHV) at BEAR beamline of ELETTRA synchrotron (Trieste, Italy). Lu 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 elsewhere¹². Lu films were deposited with a TriCon evaporation source²⁹, in which a small Ta crucible is bombarded by electrons that impinge on the crucible wall. Lu granules of 99.99 % purity from LTS Chem. Inc. were used. The crucible-sample distance was 200 mm. Deposition rate was ~ 2.5 nm/min. Film thickness was monitored with a quartz crystal microbalance during deposition. Lu films were deposited onto room-temperature substrates. A witness glass substrate was placed close to the grid-supported C film to get coated simultaneously with a similar Lu film thickness. Reflectance versus the incidence angle was measured on the witness samples at the energy of 100 eV and the angular positions of the minima and maxima were used to calculate the Lu film thickness. Since reflectance measurements were performed far from absorption edges, Henke optical constants²⁵ 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 Laboratory³⁰. The distance on the surface sample between the area of transmittance measurements and that of reflectance measurements was less than 10 mm.

2.2 EXPERIMENTAL SETUP FOR TRANSMITTANCE MEASUREMENTS

Transmittance measurements were performed at BEAR beamline with vertical exit slits 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. Above 1,200 eV an Ag filter was used. The beam cross section at the sample was about $0.7 \times 1.5 \text{ mm}^2$.

The measurements were performed in the BEAR spectroscopy chamber³¹, connected in vacuum to the preparation chamber at a pressure of $\sim 3 \times 10^{-7} \text{ Pa}$, where in-situ samples were prepared. Three C substrates were used and their transmittance was measured previously to Lu deposition. Two and four successive Lu coatings of various thicknesses were accumulated upon the first and the second substrate, respectively, without breaking vacuum. The third substrate received only one deposition of a single Lu thickness. Each sample was transferred back and forth between the deposition chamber and the measurement chamber, always under UHV, for the deposition of the successive Lu 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 cancelled by normalizing the recorded beam intensity to the mesh current. At energies below 18 eV, fluctuations were cancelled by normalization with respect to the ring current.

3. Results and discussion

3.1 TRANSMITTANCE AND EXTINCTION COEFFICIENT OF Lu

We measured the transmittance of Lu films with the following thicknesses: 24.6, 28.0, 34.5, 44.2, 71.5, 90.0, and 114.0 nm. The transmittance of the Lu films normalized to the transmittance of the uncoated substrate is plotted in Fig. 1. There are two high transmission bands peaked at $\sim 1,486$ and ~ 25 eV, right below Lu M_5 and $O_{2,3}$ edges, respectively. The low-energy band of relatively large transmittance extends within ~ 20 - 26 -eV. Close transmittance bands have been measured for other

rare earths; hence Lu, as other lanthanides such as La, Ce, Pr, Nd, Eu, Gd, Tb, Dy, Tm, and Yb, is a promising material for transmittance filters or multilayer spacers for the extreme ultraviolet in the ~12-26-eV spectral range, where there has been a lack of low-absorbing materials until recently. The slight oscillations at ~72, ~100, ~285, and ~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 Al and Si filters, to carbon contamination of the optics, and to the slight presence of O either at the optics or on the sample, respectively.

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

$$\ln\left(\frac{T_{fs}}{T_s}\right) \approx A - \left(\frac{4\pi k}{\lambda}\right) \cdot d \quad (1)$$

where T_s and T_{fs} represent the transmittance of the uncoated substrate and of the substrate coated with a Lu film, respectively; λ is the radiation wavelength in vacuum; d stands for the Lu film thickness. Eq. (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 Lu 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. The semi-empirical data of Henke^{25,30}, also plotted in Fig. 2, were calculated with a density of 9.84 g/cm³. The aforementioned presence of C, Al, Si, and O oscillations at the C K, Al L_{2,3}, Si L_{2,3} and O K edges is less significant on k than on transmittance because samples of different Lu thicknesses with similar presence of contaminants

(either on the sample or on 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.

When reflectance is not negligible, the application of Eq. (1) to calculate k through the slope of the log of transmittance versus thickness may result in uncertainties. Furthermore, in the 7.75-17.5 eV range only one sample was available and the slope method could not be used. In order to overcome this, we proceeded in an iterative way. The iterative method was applied in the 3-30-eV range. For the first iteration, initial k values were obtained using the slope method, except within the 7.75-17.5 eV range, where initial k values were obtained through a direct calculation from transmittance measurements using Eq. 1 and neglecting reflectance ($A=0$). 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 the next sub-section). Once a first set of data $\{n(E), k(E)\}$ was available, the transmittance ratio of the C/Lu 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 k data plotted in Fig. 2 were progressively modified from 11 to 3 eV in an attempt to better match literature data below 6 eV, which is explained in the next sub-section.

k values at the $O_{2,3}$ edge and around are presented in Fig. 3. The smallest value of k is obtained at ~ 25.1 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, Eu⁸ at 16.7 eV, Pr⁷ at 16.87 eV, Nd⁵ at ~ 17 eV, Tb³ at ~19.5 eV, Gd⁵ at ~19.7 eV, Dy⁴ at ~20.2 eV, Yb^{1,2} at 21.2 eV, Tm⁹ at 23 eV, and Sc¹⁰ at 27 eV. As with other lanthanides, optical properties of Lu in this range are promising for its use in transmittance filters or reflective multilayers. However, Lu, as its neighbors in the periodic table, is a reactive material, and this may result in the need to develop a protective layer. N_{4,5} edge for Lu, similar to what was obtained for Yb², is much smoother than for other lanthanides. This is probably due to the fact the outer electron shell configuration of the two elements is very similar ([Xe] 4f¹⁴ 6s² for Yb and [Xe] 4f¹⁴ 5d¹ 6s² for Lu) and then the oscillation strength of the N edge is very close. This aspect could be further investigated in theoretical literature.

k at the M_{4,5} edge is presented in Fig. 4, along with the semi-empirical data of Henke. At this energy range, the transmittance-versus-energy curves for the different samples are not fully consistent, since they do not have a common shape. The transmittance of the two thinnest films cross at two energies and the same happens with the transmittance of the two thickest films. This is attributed to the small signal available at these energies, which may be masked by scattered radiation of smaller energies, even though we used an Ag filter to reduce this effect. In fact, we extended the measurement range away from the nominal limit of 1,600 eV of the beamline. We did so in order to be able to provide optical constants on the M₄₅ edge of Lu, due to the lack of any experimental data in the literature. In the calculation of k we decided to use the measurements on all samples since we had no guide to reject any data. We slightly modified k in the 1,690-1,800 eV range in order to smoothly connect with Henke data.

3.2 REFRACTIVE INDEX CALCULATION THROUGH DISPERSION RELATIONS

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

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

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 1,800 and $3 \cdot 10^4$ eV we used Henke data from CXRO's web^{24,30}; the two sets of data were coupled with a smooth connection. For even larger energies, the calculations of Chantler et al.³² were used up to $4.3 \cdot 10^5$ 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, no direct data on k was found in the literature. We considered the papers of Schüler³³, Weaver and Lynch³⁴, and Homewood and Trodahl³⁵. The three papers gave data on reflectance and optical conductivity (or other functions from which the former ones could be immediately calculated). Unfortunately, even though the three pieces of research must have involved the calculation of also n and k , these data were not published. Other than the above literature, Pétrakian³⁶ reported the optical conductivity of Lu films on the 1.6-6.2 eV, from which k cannot be calculated. More recently, Saini et al.³⁷ used a theoretical model to calculate the optical properties of Lu; however, again, they only published optical conductivity data.

Let us see the way how we obtained the small-energy extrapolation starting with the available data. We discarded the data of Weaver and Lynch³⁴ because their work was performed with single crystals of Lu (versus thin films in the other two papers), so that their samples might have got somewhat oxidized, whereas the samples of Schüler³³ and of Homewood and Trodahl³⁵ consisted of thin films that were prepared and maintained in UHV. The calculation of n and k from reflectance and optical conductivity data from Refs. 33 and 35 involved the solution of a 4th order algebraic equation, from which only two of the four solutions could be immediately discarded because they were complex

numbers. The other two solutions looked acceptable in principle. Unfortunately, the data from Refs. 33 and 35 resulted in non-coincident solutions of n and k , so that we had as many as 4 choices of k for our low energy extrapolations. We finally chose a specific solution for each of the two selected papers which better matched each other and better connected with our data, even though this connection was not smooth; unfortunately, the data of Weaver and Lynch³⁴ resulted in two other solutions which were not coincident with the selected ones from Refs. 33 and 35 and were of no help for the selection of data. The data of Homewood and Trodahl matched better our data than Schüler's data did, and hence the former was initially selected as a continuation of our data³⁸. As a help to make the good choice, those data were compared with n and k data of neighbors in the periodic table: Tm⁹ and Yb². In order to enable a smooth connection of our data with Ref. 35, both were modified in the 3-6 eV range. Homewood and Trodahl's data only extended down to 1.5 eV; therefore, Schüler's k data were used below this energy down to 0.28 eV. The extrapolation to zero energy was performed by fitting a Drude model on Schüler's data.

The refractive index n obtained with Eq. (2) on the k data gathered in the whole spectrum was rather different than the data that we calculated from Schüler³³ and Homewood and Trodahl³⁵ data in the coincidence range. Since we did not have any reason to trust more k than n data of Refs. 33 and 35, we decided to use both sets of data in our construction of k data in the whole spectrum by proceeding in the following way. The refractive index n calculated as said above was modified by replacing the 0.28-6 eV range with Schüler's³³ and Homewood and Trodahl's³⁵ n data. The data between 6 and 11 eV were modified to get a smooth connection with the refractive index obtained with Eq. 2. This new and complete set of n data was used in an inverse KK analysis:

$$k(E) = -\frac{2E}{\pi} \text{P} \int_0^{\infty} \frac{[n(E') - 1]}{E'^2 - E^2} dE', \quad (3)$$

so that a new set of k data was obtained. Then we had two sets of k data that were similar for energies above 11 eV, but were different below this energy. An average set of k data was obtained between the two sets, and the latter was assumed to be a better solution than the first set because it involved both n and k data (versus only data of k) of Lu obtained from the literature.

Fig. 5 displays k data of Lu obtained in the present research along with literature data, calculations, and extrapolations that were gathered for KK analysis.

Fig. 6 displays $\delta=1-n$ calculated with Eq. (2) using the data plotted in Fig. 5; n and δ at O_{2,3} and M_{4,5} edges are shown in Figs. 7 and 8, respectively. Only Homewood and Trodahl data³⁵ or the semi-empirical data of Henke are available for comparison.

3.3 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 global accuracy of k data. It is useful to define the effective number of electrons per atom $n_{eff}(E)$ contributing to k up to given energy E :

$$n_{eff}(E) = \frac{4\varepsilon_0 m}{\pi N_{at} e^2 h^2} \int_0^E E' k(E') dE' \quad (4)$$

where N_{at} is the atom density, e is the electron charge, ε_0 is the permittivity of vacuum, m is the electron mass, and h is Planck's constant³⁹. The f sum rule expresses that the high-energy limit of the effective number of electrons must reach $Z=71$, i.e., the atomic number of Lu. When the relativistic correction on scattering factors is taken into account, the high-energy limit of Eq. (4) is somewhat modified. The following modified Z was adopted here: $Z^*=69.70$ ⁴⁰. The high-energy limit that we obtained by integrating the data set plotted in Fig. 5 using Eq. (4) was 70.77, which is only a 1.5% larger than the above Z^* value. The main contribution to n_{eff} was found to come from the ~ 1 to 6×10^5

eV range. The small difference with Z^* may come from 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 KK analysis is obtained with the inertial sum rule:

$$\int_0^{\infty} [n(E) - 1] dE = 0 \quad (5)$$

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. (5)³⁹ is:

$$\zeta = \frac{\int_0^{\infty} [n(E) - 1] dE}{\int_0^{\infty} |n(E) - 1| dE} \quad (6)$$

Shiles et al.³⁹ suggested that a good value of ζ should stand within ± 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 rule test is well within the above top value, which, along with the above obtained result for the f sum rule, suggest good consistency of n and k data.

CONCLUSIONS

The transmittance of thin films of Lu deposited by evaporation has been measured in situ in the 3-1,800 eV photon energy range under UHV conditions. The extinction coefficient k of Lu has been calculated from transmittance measurements in the same spectral range. Lu features an absorption minimum at ~25.1 eV. This relatively low absorption at this spectral range makes Lu a promising candidate for transmittance filters and reflective multilayers. Given the reactivity of Lu, as with other lanthanides, a surface passivation method is expected to be required to prevent surface instability of Lu in contact with atmosphere.

The refractive index n of Lu in the same range was obtained with KK analysis over an extended spectral range. A novel way of extending our data with literature data was devised by using both n and k (versus only k data) in our k extrapolation; n literature data were used to modify the data obtained by KK analysis, which by inverse KK analysis provided a second estimate of k , which was averaged with the initial one.

Current data on Lu are the first reported experimental data of both the extinction coefficient and the refractive index in the whole range. The current data encompass Lu 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 Lu.

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38. Once the described calculation and selection process had been performed, in a private communication Prof. J. Trodahl provided us with n and k data for Lu that they had obtained in their research (Ref. 35). Those data resulted to be coincident with the solution that we had previously calculated and selected.
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Figure captions

Fig. 1. (color online) The transmittance of Lu films with various thicknesses normalized to the transmittance of the substrate versus the logarithm of photon energy.

Fig. 2. (color online) Log-log plot of the extinction coefficient of Lu as a function of photon energy, along with the data of Homewood and Trodahl³⁵ and the data of Henke *et al.*²⁴

Fig. 3. (color online) The extinction coefficient of Lu as a function of photon energy at the small energy range, along with the data of Homewood and Trodahl³⁵, and the data of Henke *et al.*²⁴

Fig. 4. (color online) The extinction coefficient of Lu versus photon energy at the M_{4,5} edge, along with the data of Henke *et al.*²⁴

Fig. 5. (color online) Log-log plot of k data that map a wide spectral range using the current data along with the data of Schüler³³, Homewood and Trodahl³⁵, Henke *et al.*²⁴, and Chantler *et al.*³², and extrapolations in the two extremes.

Fig. 6. (color online) Log-log plot of $\delta=1-n$ versus photon energy. The data of Henke *et al.*²⁴ are also represented

Fig. 7. (color online) n versus photon energy at the low energy range. The data of Homewood and Trodahl³⁵ and of Henke *et al.*²⁴ are also represented

Fig. 8. (color online) $\delta=1-n$ versus photon energy at the M_{4,5} edge. Henke data²⁴ are also represented.

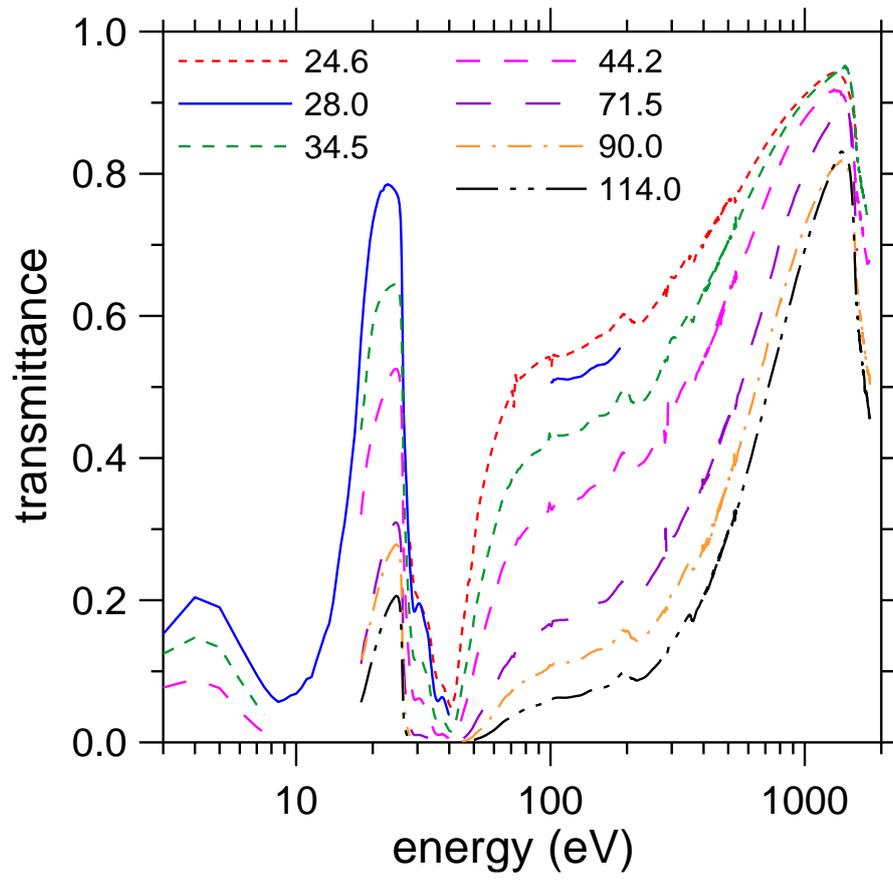


Fig 1

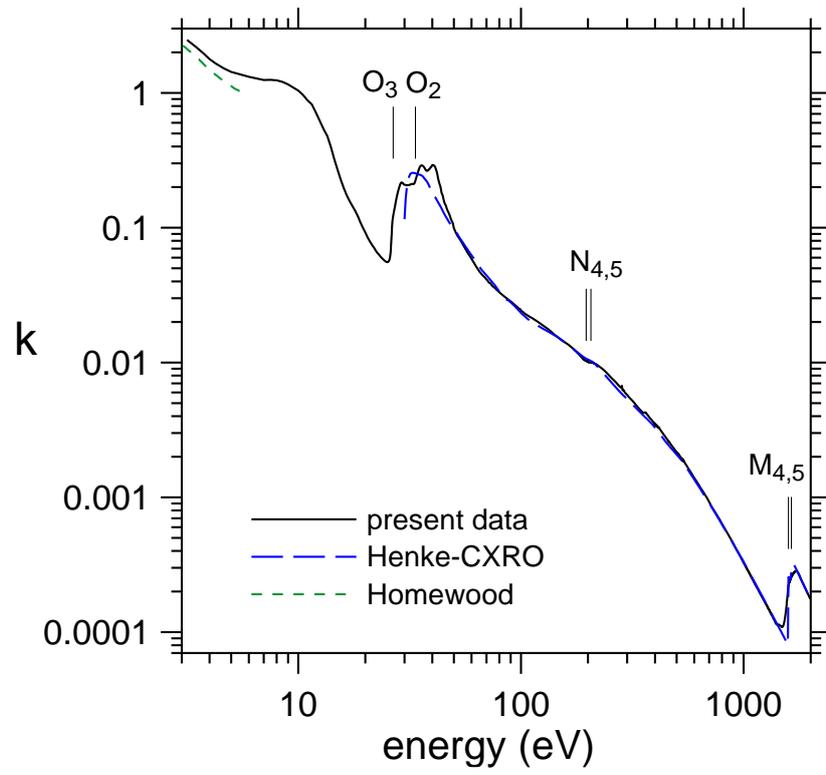


Fig 2

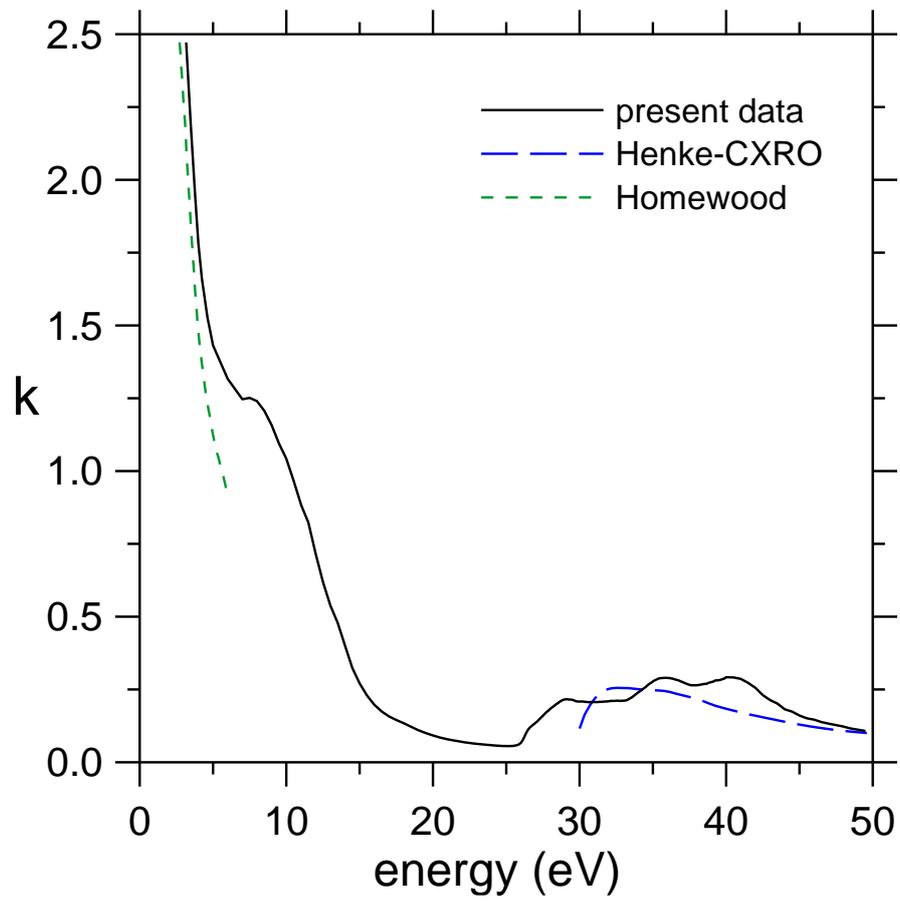


Fig 3

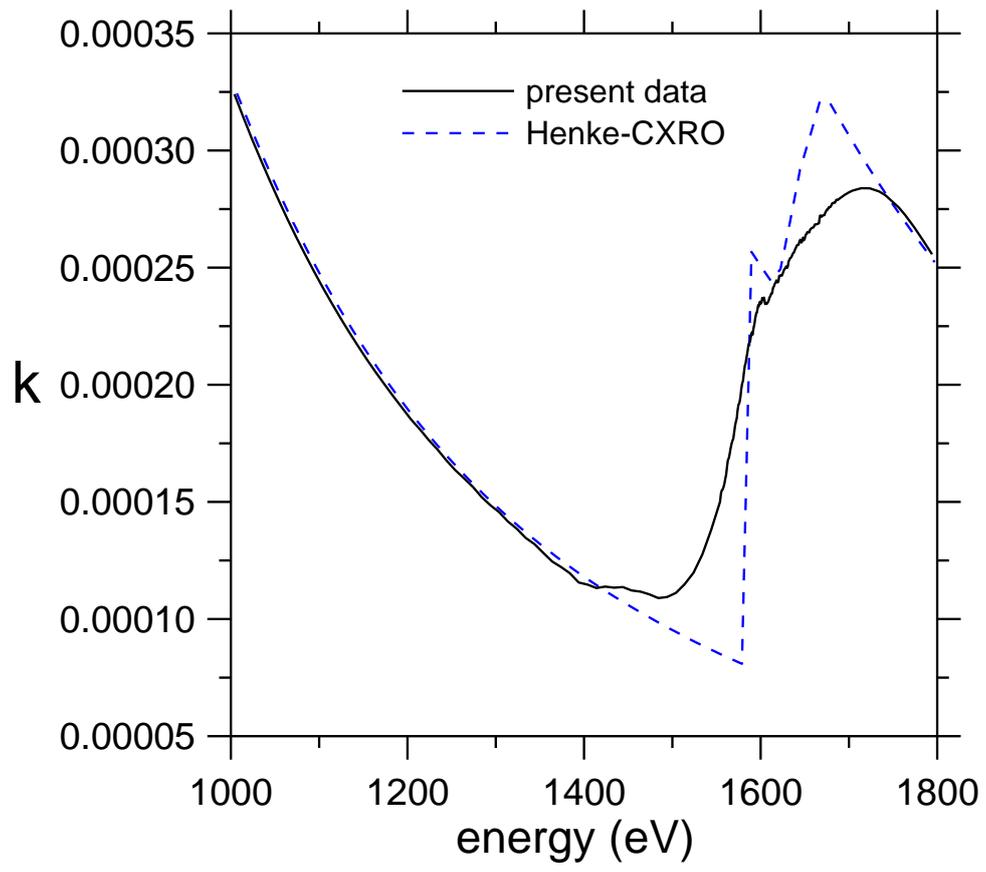


Fig 4

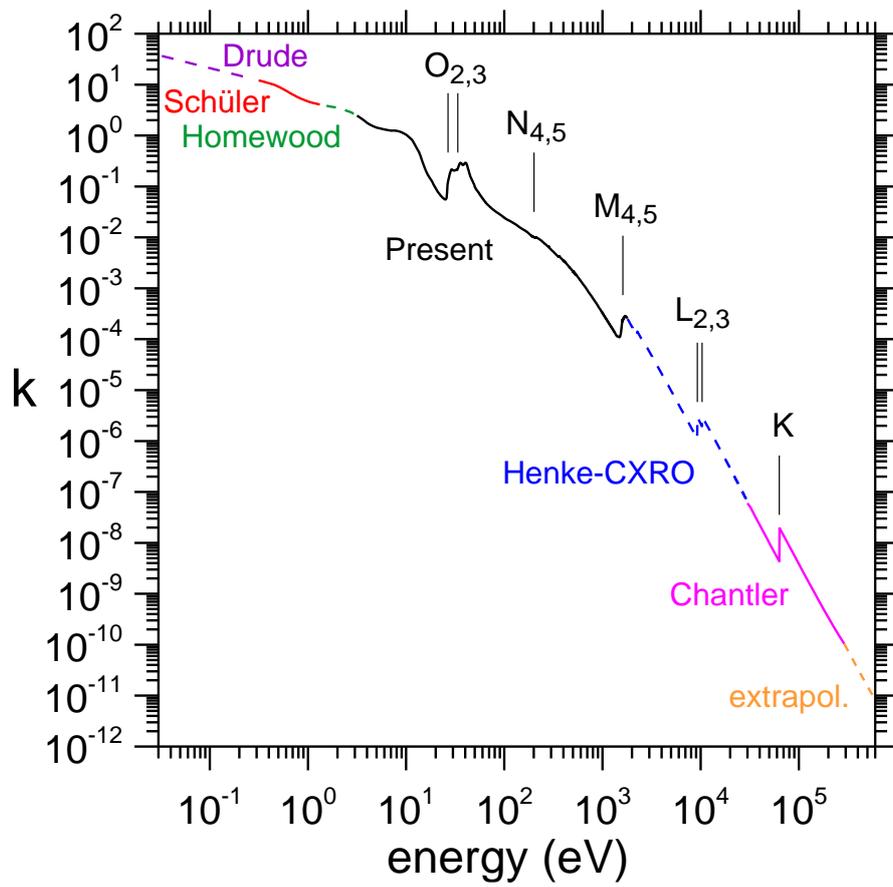


Fig 5

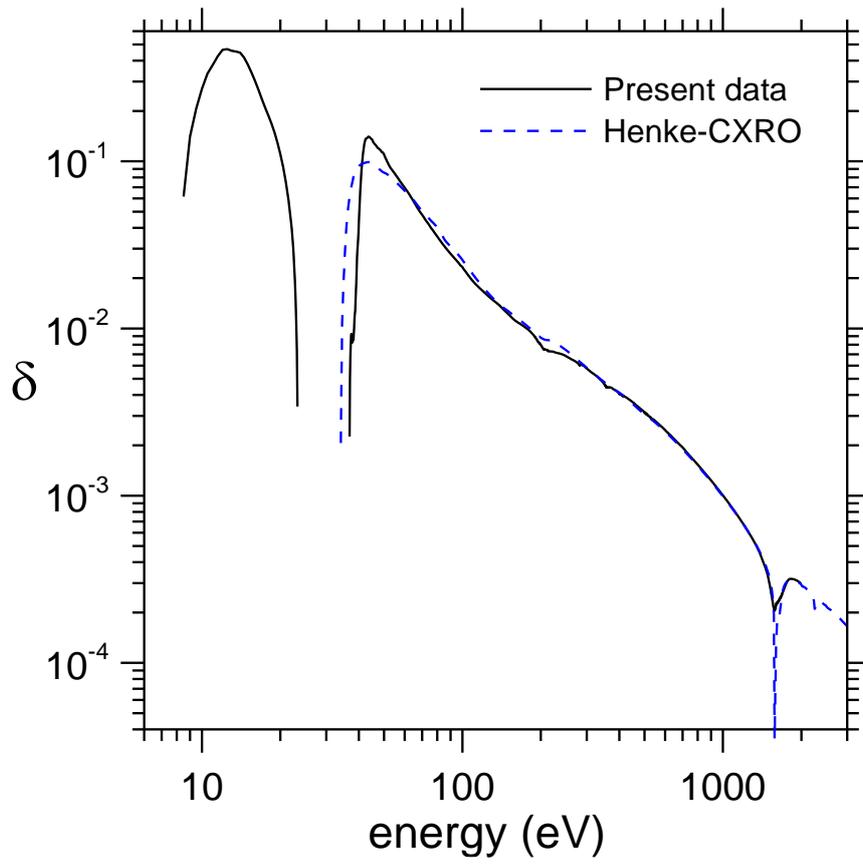


Fig 6

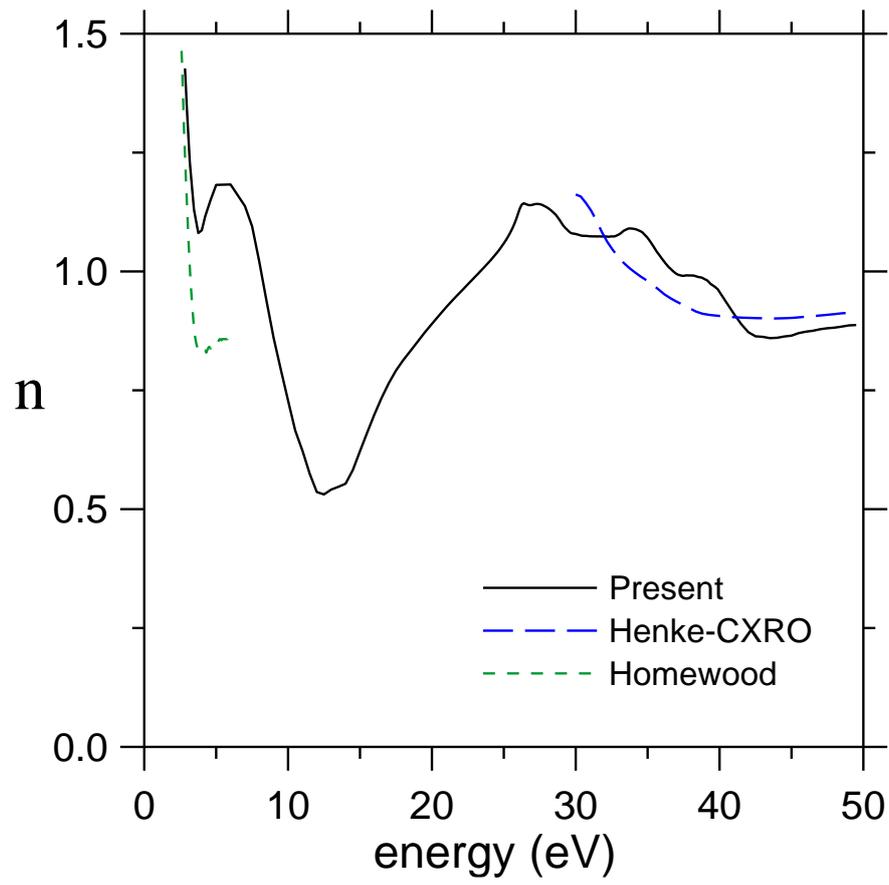


Fig 7

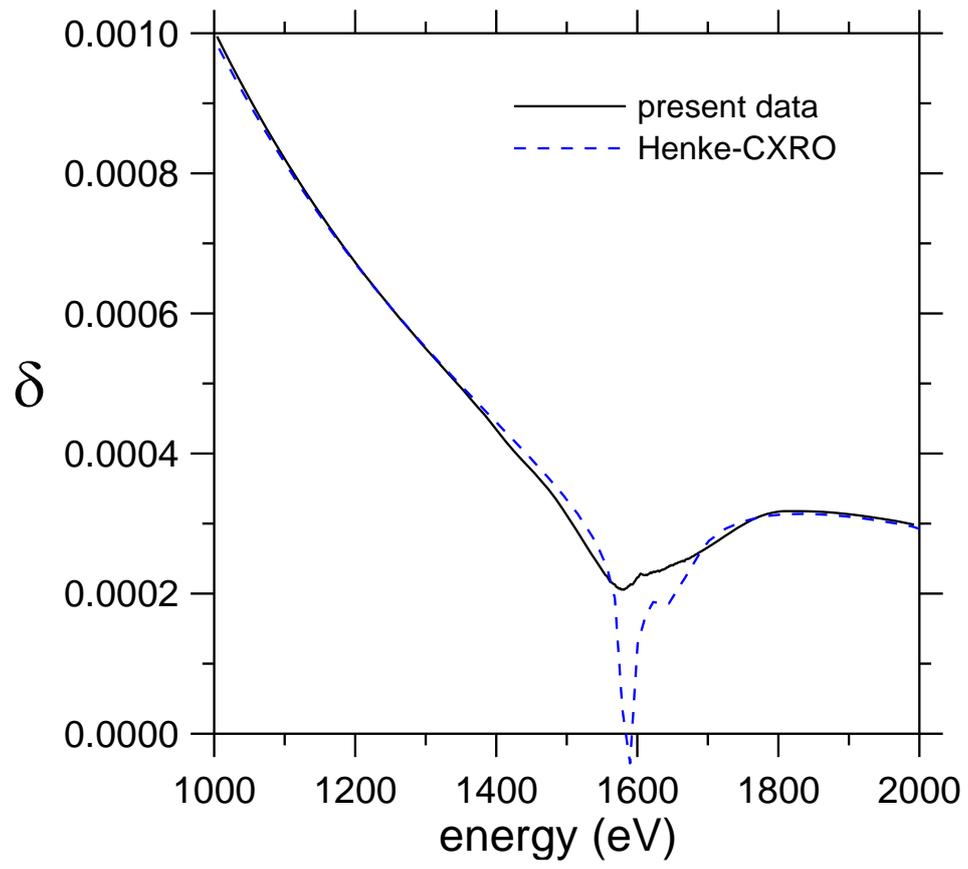


Fig 8