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Investigation of Inner Shell Excitations in Solids by Synchrotron Radiation

by

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INVESTIGATION OF INNER SHELL EXCITATIONS IN SOLIDS BY SYNCHROTRON RADIATION [+)]

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I. INTRODUCTION

The optical properties of many gases and solids are well known in both the photon energy range below 10 eV (infrared, visible and near ultraviolet) and in the X-ray region. However, twenty years ago only a few data on optical properties were available in the intermediate range, in the vacuum ultraviolet or soft X-ray region because of great experimental difficulties. Since then this situation has been improved thanks to the development of new vacuum spectrometers, radiation detectors and light sources. One of the most important steps was the introduction of synchrotron radiation as a light source for this spectral range.

Synchrotron radiation is emitted by electrons radially accelerated in the magnets of high energy electron synchrotrons or storage rings. The general features of synchrotron radiation may be found in several textbooks on classical electrodynamics[1], more detailed information may be obtained from the paper of Schwinger[2] and the book by Sokolov and Ternov[3].

The spectrum of synchrotron radiation emitted by running high energy synchrotrons and storage rings (electron energy 10^8 eV to 10^{10} eV, circulating current 1 mA to 100 mA) spans the whole range from the soft X-ray region to the infrared. The intensity in the vacuum ultraviolet is higher than that of any conventional light source. The spectral distribution is continuous and smooth. The intensity is concentrated in a small cone tangential to the electron orbit. Further advantages are the high degree of polarization and the fact that the source works in a very good vacuum ($<10^{-6}$ Torr).

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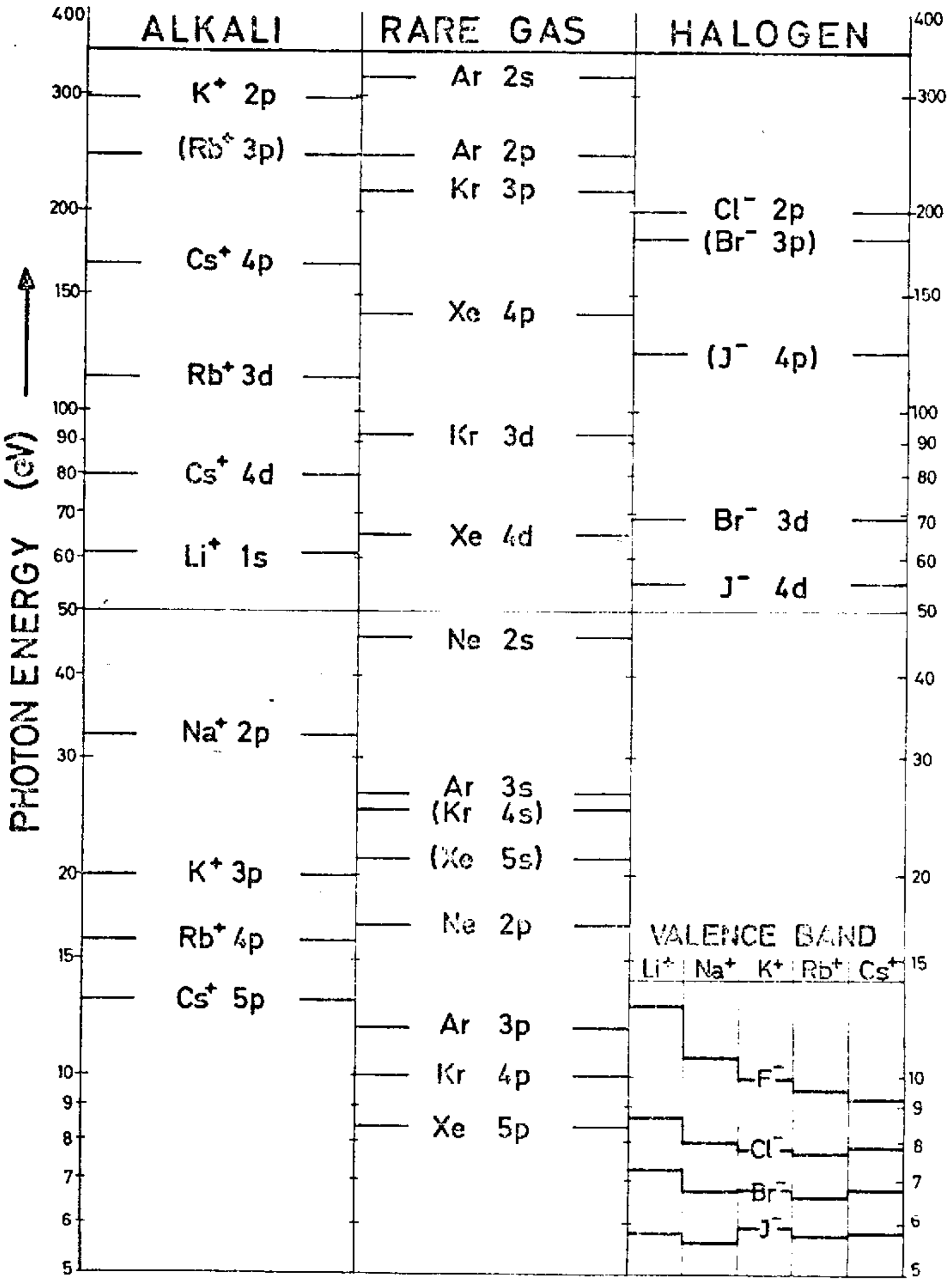

Fig. 1 Threshold energies for transitions from the different occupied shells in the alkali halides and rare gases

The first attempts to use synchrotron radiation for optical experiments in the extreme ultraviolet were made by Hartman and Tomboulian[4] as early as 1953, but systematic use for spectroscopic purpose only began after 1960. Several papers[5-7] may be consulted for more detailed information on the state of spectroscopic work with synchrotron radiation at different accelerators. In addition to the synchrotrons and the storage rings which have been used so far (Cornell, NBS, Frascati, Tokyo, Glasgow, Bonn, Moscow and DESY synchrotrons and the University of Wisconsin storage ring) new facilities will be opened in the near future (Daresbury synchrotron and storage rings in Tokyo, Orsay, Stanford and Hamburg). Nothing will be said here on experimental techniques since they are, essentially, all reviewed in Samson's book[8].

The aim of spectroscopic measurements is to obtain information on the electronic structure of solids. With conventional sources ranging up to ∿15 eV mostly electronic excitations from the valence band can be studied. For metals and low band gap semiconductors transitions to states several eV above the bottom of the conduction band can be reached. For wide band gap materials such as alkali halides and solid rare gases only the first excitations can be studied; in solid Ne and He they even lie above that limit. In the energy range above 15 eV transitions from tightly bound core levels to unoccupied states above the Fermi level give the main contribution to the optical absorption. These core states are very well localized and the wave functions of neighbouring atoms do not, therefore, overlap. As a result, no k-dependence of the energy is observed.

The width of core levels is determined by lifetime broadening (Auger effect) but for levels with excitation energies in the extreme ultraviolet this broadening is much smaller than for those in the X-ray region.

The optical spectra in the extreme ultraviolet are different from those found in the fundamental absorption or X-ray region. On the following pages we are going to review some of their main characteristics.

II. ONE ELECTRON MODEL

A. Energy Levels

The electronic structure of atoms and solids can be described in a first order approximation by the one electron model or by the energy band scheme resp. Optical measurements yield information on the position of the tightly bound initial states and the final states (in solids the positions of the energy bands) with respect to the Fermi energy. Fig. 1 shows the onset of absorption due to the excitation of electrons from the different subshells for the alkali halides and (solid) rare gases. Most of the positions have been established by optical experiments; the positions of energy levels in brackets are taken from the tables of Bearden and Burr[9], where the atomic energy levels of the other elements are also listed.

For an experimentalist the first step is to compare his experimental curves with theoretical energy level diagrams, taking into account the optical selection rules. Because of the small width of the initial state the spectra should reflect the structure of the conduction bands. Many attempts have been made to interprete the experimental results by assigning the absorption maxima to electron transitions from the flat core band to the different conduction bands at points of high symmetry or at distinct regions of the Brillouin-zone. However, in most cases the knowledge of the energy band scheme alone cannot lead to an unambiguous interpretation and thereby to an understanding of the spectra above threshold.

B. Density of States

The optical absorption of solids is proportional to ε_2 as given in the following relation:

$$\varepsilon_2 = F \int \frac{M(E,k)^2}{|\nabla_K(E_f - E_i)|} \, dS$$

M(E,k) is the transition matrix element between initial state E_i and final state E_f. For transitions from core states $\nabla_K E_i$ is equal to zero. Furthermore, we assume that M(E,k) = const. In this case ε_2 is proportional to the density of states of the conduction band.

We are now going to compare some experimental data with theoretical calculations. Generally, this can only be done for a range of several eV above threshold which is covered by the energy band calculations. Our comparison will be made for some transitions in the alkali halides and solid rare gases. In the core transitions of the solid rare gases detailed fine structure has been found for Xe 4d[10,11], Kr 3d[11] and Ar 2p[12] excitations. Rössler has calculated the energy bands and density of states curves of the solid rare gases (Ne, Ar, Kr and Xe) and has shown that the major part of these fine structures can be explained as being caused by density of states effects[13,14]. Fig. 2 shows the experimental curve of the 4d transitions in solid Xe[10,11] together with the density of states curve, taking into account the spin orbit splitting energy of the 4d shell of 2 eV and the degree of degeneracy of the two subshells[14]. The first peak in the density of states curve has been energetically adjusted to the first broad and pronounced peak D in the experimental curve. After doing so we see that the energy position of most of the maxima of both curves coincide. Furthermore, the shapes of both curves are similar. Deviations can be seen at peak F' and around H

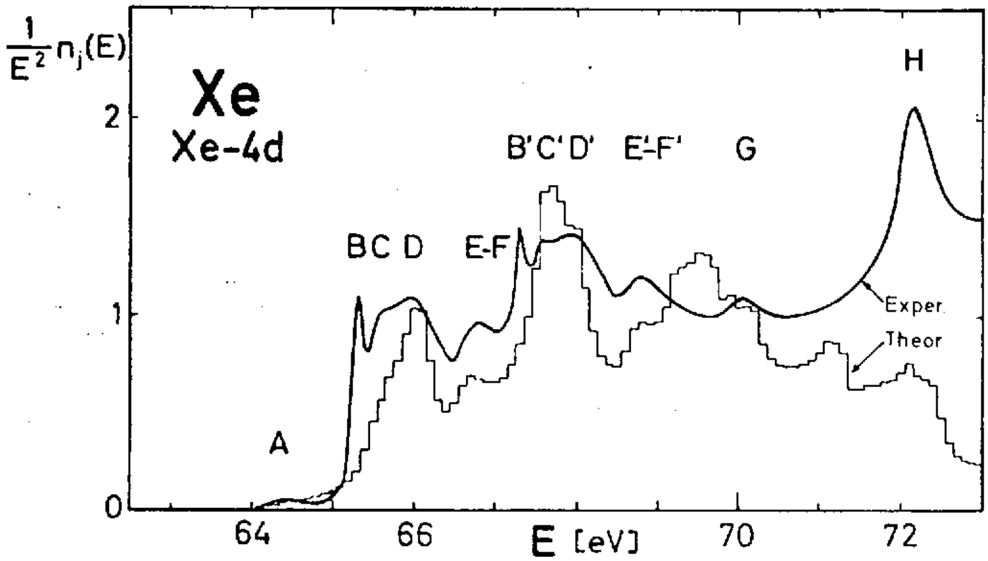

Fig. 2 Absorption spectrum from 4d states and joint density of states in solid Xe (fig. 4 of ref. 14 improved by Rössler)

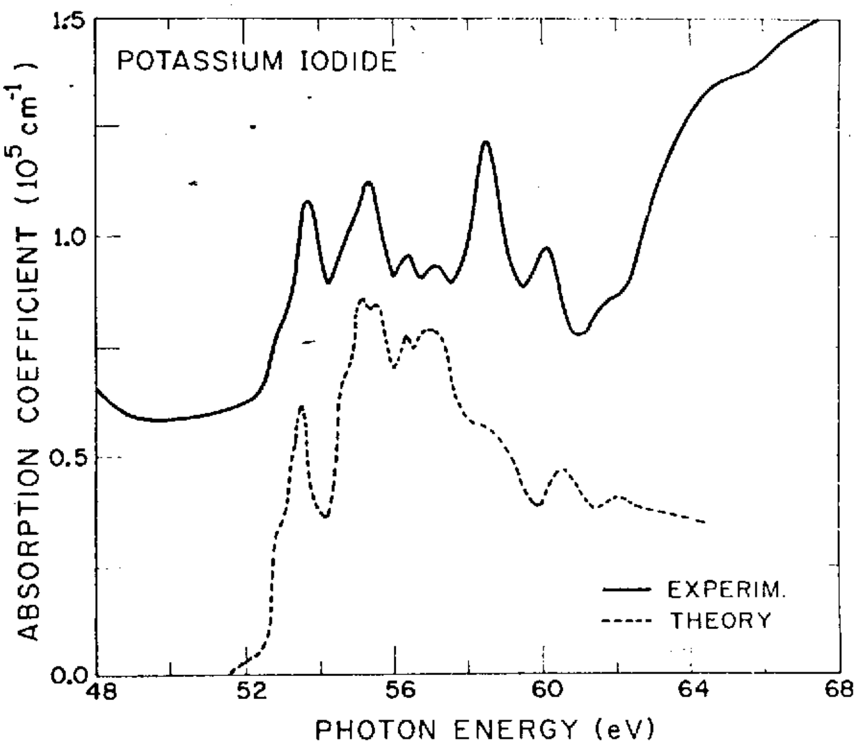

Fig. 3 Absorption spectrum from I^- 4d states and joint density of states in KI (fig. 13 of ref. 15)

where the experimental curve increases, whereas the theoretical curve decreases. The peaks B and B' are not described by the density of states curve. Their width is smaller than that of density of states peaks. We will come back to these extra-peaks later.

The 4d absorption spectrum of the isolectronic I^- ion in KI is shown in Fig. 3 together with the density of states curve[15]. As for Xe, this curve has been constructed by the density of states of the conduction band, taking into account the spin orbit splitting of the 4d level and the statistical weight of the two subshells. We find an almost one-to-one correspondence of the energy positions of the maxima of both curves. The shapes of both curves are similar at the onset but show marked differences at higher energies. The "extra lines" B and B' found for Xe have no counterpart in KI.

A slithly better agreement between the experimental and the density of states curve has been found for the Kr 3d[14,16] and Ar 2p[14] spectra. The Ar 2p spectrum will be shown in the next chapter (Fig. 6).

These examples show that many of the features of the spectra can be explained by the density of states of the conduction band. There are, however, still many discrepancies between theory and experiment which indicate that the spectra cannot be completely understood in terms of this simple model.

In optical experiments one can derive the optical constants ε_1 and ε_2 from the experimental results, but they do not directly yield the distribution of the density of states. For a better test of the validity of the one electron approximation for solids it is necessary to construct theoretical ε_1 and ε_2 curves from the theoretical density of states curves including the transition matrix elements. Unfortunately, the bulk of band calculations was made without the extension to density of states curves and to the authors' knowledge no case is known, where a complete ε_2 curve for core transitions has been calculated.

C. Continuum Absorption

For transition into states far above threshold almost no band calculations are available for a comparison with the experiments. For this case, however, atomic models, in the simplest case one electron models, can be used which describe atomic absorption far above the ionization limit. In the vacuum ultraviolet many cases are known where the absorption behaviour strongly deviates from the hydrogen case. For a pure Coulomb potential the absorption coefficient shows a saw-tooth-like behaviour typical in the X-ray region. The absorption increases step-like at threshold and decreases above treshold with increasing photon energy E according to $E^{-\alpha}$ (α being constant).

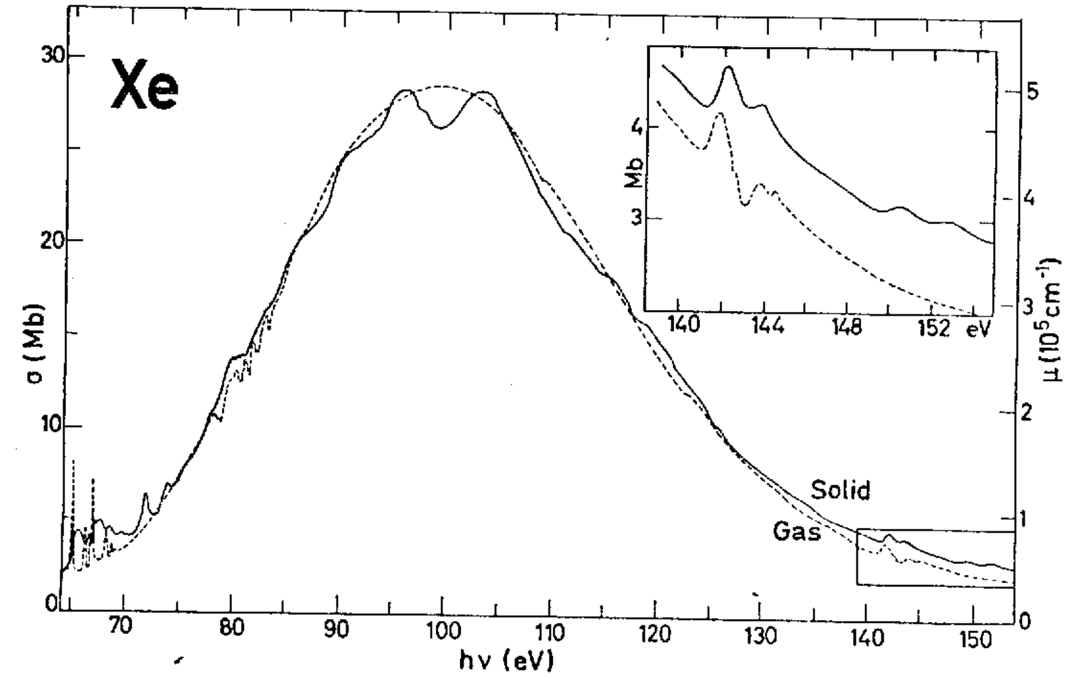

Fig. 4 Absorption spectrum of solid (solid curve) and gaseous (dotted curve) Xe in the range of 4d transitions (ref. 11)

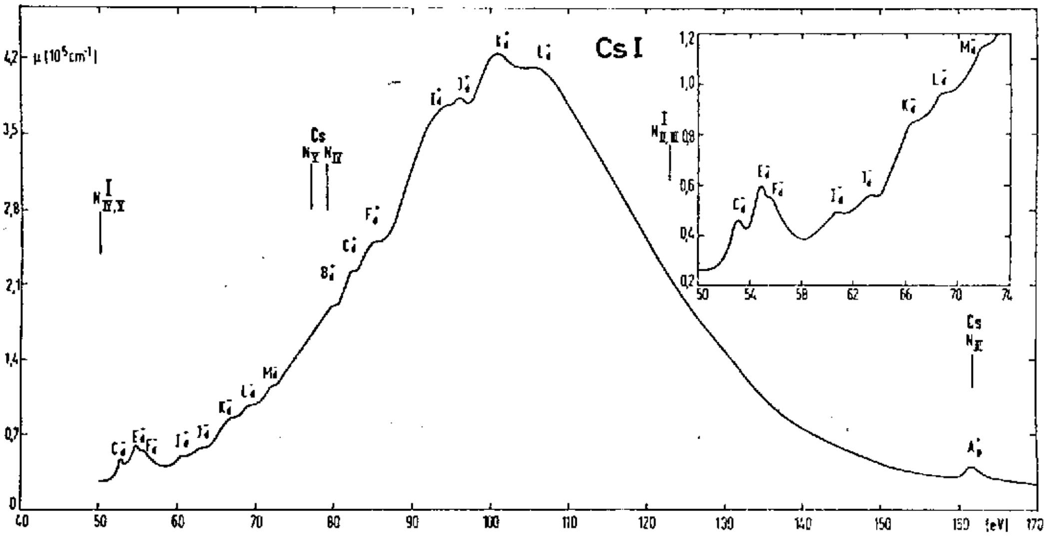

Fig. 5 Absorption spectrum of CsI from 50 to 170 eV (ref. 22)

An example of a non-hydrogenic absorption behaviour can be seen in the 4d continuum absorption of gaseous and solid Xe (Fig. 4). Above the fine structure near threshold we see a broad absorption maximum with its peak near 100 eV, i.e. 35 eV above threshold. This behaviour was observed in atomic Xe for the first time by Ederer[17] and explained by Cooper[18] as a delayed onset of d→f transitions which are suppressed near threshold. The reason for this suppression is a potential barrier around the nucleus resulting from superposition of the effective Coulomb potential and the $\ell(\ell+1)/r^2$ pseudopotential in the radial part of the Schrödinger equation[19]. On the basis of a realistic potential the absorption cross-sections have been calculated for the rare gases and for many other elements[20,21]. The results describe, at least qualitatively, the main features of continuum absorption.

A comparison of the experimental results for the continuum absorption of the rare gases shows excellent agreement of the continuum absorption cross section for both the atomic and solid state[11,12]. This strongly supports the application of this one electron atomic theory for the explanation of continuum absorption of solids. Similar characteristics in the absorption have also been found in some alkali halides[15,22] (see Fig. 5 for CsI), some semiconductors[23], almost all lanthanides[24,25], and many metals[26,27].

III. BEYOND THE ONE ELECTRON MODEL

A. Coulomb interaction between hole and electron

We are now going to discuss the origin of the "extra lines" B and B' in the 4d absorption spectrum of solid Xe (Fig. 2). A similar structure can be found in the 2p absorption of solid Ar (Fig. 6) where peak A is obviously not described by the density of states curve. The energy positions of the lines in Xe and Ar (as well as corresponding lines in the Kr 3d absorption) are very close to the first absorption lines in the gas. This leads to the assumption that they are due to the formation of Frenkel excitons[28]. In all solid rare gases and most of the alkali halides the wavefunctions near the bottom of the conduction band have s-symmetry. According to Rössler, therefore the exciton in Ar with its hole in a p-symmetric state lies below the onset of interband transitions, whereas for Xe and Kr with a d-symmetric initial state the excitons are above the onset. As a result, they can decay into continuum states and are, therefore, very weak in contrast to the exciton line in Ar whose oscillator strength is comparable to that of the corresponding gas line.

Whereas the exciton line in Xe is very weak it completely disappears in KI[15]. For Ar 2p-absorption a corresponding case in the

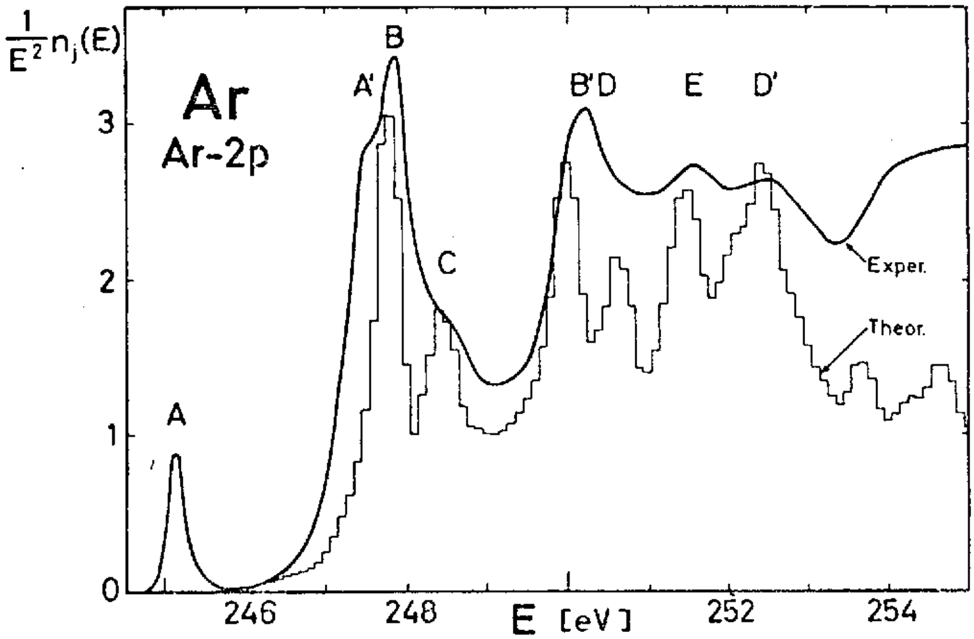

Fig. 6 Absorption spectrum from 2p states and joint density of states in solid Ar (fig. 6 of ref. 14)

alkali halides is the Cl^- 2p absorption which has been discussed for NaCl by Lipari and Kunz[29]. The authors believe that this spectrum can also be entirely explained by density of states effects. It should, however, be noted that the width of the first peaks in the experimental curve is obviously due to the instrumental energy resolution which becomes worse for higher energies. The same is true for the Ar 2p curve (Fig. 6) where the exciton line A has the intrumental width, and could possibly be otherwise smaller than the density of states peaks of the theoretical curve. As for NaCl only three peaks, two of which are spin orbit mates, have been compared, a final statement cannot be made at present. The difference in the oscillator strength of the exciton lines in Xe and Ar makes one believe that the absence of exciton lines in the I^- 4d spectrum must not necessarily also lead to the absence of exciton lines in the Cl^- 2p spectrum.

So far, exciton effects have mainly given rise to additional absorption lines. The rest of the absorption features can be explained as being due to density of states effects; however, the agreement between theory and experiment is not so good that an additional influence of Coulomb interaction onto the shape of the interband absorption lines can be excluded.

We are now going to discuss core excitation spectra with excitation energies below 50 eV. As can be seen from Fig. 1 the excitation energy for the outermost alkali ion shells lies at ∿13 eV for Cs^+ 5p, ∿15 eV for Rb^+4p, ∿20 eV for K^+3p, and ∿30 eV for Na^+2p. For these transitions reflection[30-32], absorption[33-36], and photoemission[37-40] have been studied. The spectra show sharp structures near the onset of transitions which are very close to the energy position of the first excitation in the free ion[41]. This and the energy distribution of the emitted photoelectrons is a strong support for the assumption that the lines are of excitonic origin.

In the valence band spectra as well as in the spectra of transitions from the outermost shells of the alkali ions the sharp exciton lines are followed by broader structures which can be interpreted as interband transitions. Here the comparison with the pure one electron density of states curves shows considerable disagreement. It turns out that here Coulomb interaction also drastically influences the spectral shape above threshold[16].

B. Exchange interaction, multiplet splitting, configuration interaction and autoionization

We have already mentioned the 4d continuum absorption maximum in the lanthanides. Below this d→f maximum we find an area of fine structure near threshold[25,42] (at ∿110 eV) which extends over an energy range of ∿20 eV. These fine structure absorption lines have been ascribed to transitions from the 4d shell into the unoccupied states of the 4f shell[42]. This assumption is supported by the fact that the absorption spectra of the metals and their oxides are very similar[25]. In an energy band scheme these unoccupied 4f levels should be close together so that no explanation for the spread of fine structure over 20 eV can be given from this picture. Dehmer et al.[43] have calculated the splitting of the $4d^9 4f^2$ configuration for Ce. The levels of this configuration are spread over a range of $\geq$ 20 eV, primarily by the effect of exchange interaction between the 4f electrons and the 4d vacancy. Dehmer et al.[43] also calculated the line intensities and obtained surprisingly good agreement with the experimental results. Both experimental and theoretical results for Ce are given in Fig. 7.

The calculations show that most of the oscillator strength is concentrated in transitions to the higher levels of the configuration. These levels, lying far above threshold and, therefore, strongly broadened by autoionization, give rise to the broad maximum ∿20 eV above threshold. Similar results have been obtained for other lanthanides[44]. The total oscillator strength of the lines is proportional to the number of 4f vacancies. This explains the disappearance of the absorption peaks upon filling the 4f shell.

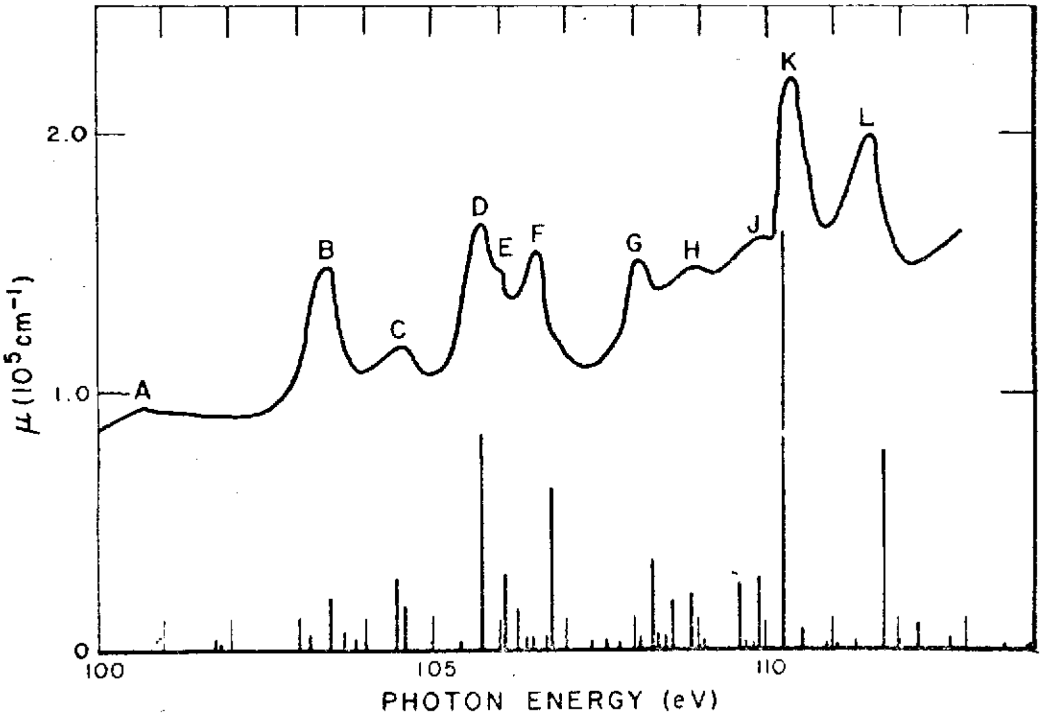

Fig. 7 Comparison of μ, the measured absorption coefficient for Ce in the region of the $N_{IV,V}$ edge with calculated relative positions and line strengths of $4d^{10}4f^{1} \rightarrow 4d^{9}4f^{2}$ transitions (the broad d→f absorption maximum about 125 eV is not included)

Dehmer et al.[43] pointed out that this interpretation of rare earth spectra may extend to any optical transition whose final state critically depends on a centrifugal barrier. This explanation should hold especially for the 3p absorption of transition metals in the third series[26] and the 5p absorption of the transition metals in the fifth series[27]. These spectra show, analogous to the 4d rare earth spectra, broad absorption maxima extending over an energy range of ∿20 eV above threshold. In contrast to the rare earth metals no marked fine structure can be seen at the onset. The characteristic features of the transition metals spectra result from 3p→3d or 5p→5d transitions resp. The width of the broad maximum above threshold is much larger than the width of the empty d-band given by band calculations.

Besides the broadening of absorption lines autoionization processes may also give rise to characteristic asymmetric line shapes. Asymmetric lines have been observed in the 2s absorption of Ne[45] and so-called window lines in the 3s absorption of Ar[46]. In the solid rare gas spectra[47] these lines are broadened and slightly

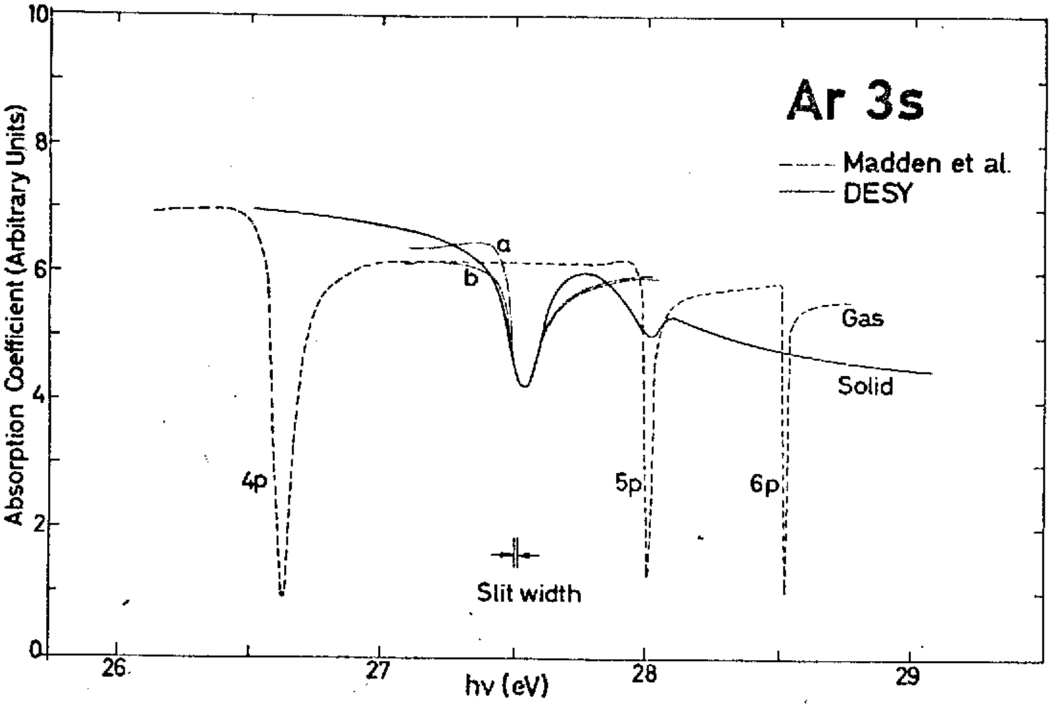

Fig. 8 3s Absorption of gaseous and solid Ar (ref. 47)

shifted to higher energies but the characteristic line shapes remain unchanged. The epxerimental results for the 3s absorption of gaseous and solid Ar are shown in Fig. 8.

Onodera[48] recently discussed the line shape problem for the interference of an exciton with continuum absorption. For the fundamental absorption spectrum of NaBr he showed that the line shape of exciton lines can only be explained by taking configuration interaction into account.

C. Multiple Excitations

Two electron excitations have been discussed for inner shell absorption of solids in different cases. In the Xe 4d spectrum (Fig. 4) near 80 eV in the gas very weak absorption peaks are observed which have been ascribed to the simultaneous excitation of a 4d and 5p electron[49]. In the same range weak structures are also observed in solid Xe[11]. The assumption that these peaks are also due to double excitations is supported by results from measurements on solid rare gas alloys[50]. Simultaneous excitations of one core electron and one valence band electron have been discussed in connection with several alkali halide absorption measurements[22,36,51]. Theoretical predictions for the oscillator strength of double excitation have, so far, given different results and were restricted to the fundamental absorption region[52,53].

Amusia et al.[54] have shown that multiply excited configurations are important in calculating the photoionization cross-sections of the two outer shells of Ar, Kr and Xe. Whereas the one electron model gives a good qualitative description of the absorption spectra, the calculated cross-sections, taking into account multi-electron correlations, are in good agreement with the experiments.

Relatively broad, periodic structures have been observed above the 2p-absorption threshold in the light metals Na, Mg and Al[55] and also in inner shell absorption spectra of some alkali halides[56-58]. These structures have been interpreted as being due to the simultaneous excitation of one electron and one or several plasmons. No theoretical calculations on the probability of such processes are available.

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