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Publication Date

1976-06-01

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Submitted to Physical Review B

LBL-5148
Preprint c. |

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X-RAY PHOTOELECTRON SPECTROSCOPIC STUDIES OF THE ELECTRONIC
STRUCTURE OF TRANSITION METAL DIFLUORIDES*

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ABSTRACT

The valence-electron densities of states of the 3d transition metal difluorides MnF_2 , FeF_2 , CoF_2 , NiF_2 , and ZnF_2 were obtained by means of high-resolution X-ray photoemission spectroscopy (XPS). Except for NiF_2 , single-crystal samples were cleaved and studied in ultra-high vacuum. With the aid of previous XPS results from alkali fluorides, the partial 3d densities of states were derived, using the constancy of the $\text{F}2s\text{-F}2p$ energy separation and relative intensity ratio. The results are in very good agreement with recent multi-configurational Hartree-Fock calculations (MCHF) by Viinikka and Bagus. The 3d and $\text{F}2p$ bands are concluded to be largely decoupled, and these results support the analysis of partial densities of states by separating the $\text{F}2p$ structure on the basis of the LiF spectrum.

* This report was done with support from the U. S. Energy Research and Development Administration. Any conclusions or opinions expressed in this report represent solely those of the authors and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the U. S. Energy Research and Development Administration.

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I. Introduction

Transition-metal compounds are of great scientific and technological interest, because they possess a wide range of interesting magnetic and electrical properties. In this paper we report a high resolution X-ray photoemission (XPS) investigation of several 3d transition-metal difluorides, MnF_2 , FeF_2 , CoF_2 , NiF_2 , and ZnF_2 .

The 3d-metal compounds have spatially localized 3d bands near the Fermi level. It is the d electrons that give rise to the interesting and wide range of properties which characterize these compounds.¹⁻⁴ Because the orbitals in 3d bands do not have a very large spatial extent beyond the ionic cores, they are not broadened very much by nearest-neighbor overlap. Thus the d-bands are narrow and atomic-like. Photoemission studies of the 3d bands are of particular interest theoretically because the usual band-structure methods that work well for other solids fail to deal with the highly localized (correlated) nature of the 3d electrons.^{6,7} However, simple atomic models have not been completely successful in explaining the properties of these materials, either.

We selected the 3d metal difluorides for study because they are the most ionic and, among the simplest of 3d-metal compounds. They are also the most stable, an important factor experimentally. Their electronic structures may provide fiducial marks in understanding the 3d bands in more complicated, more covalent compounds. These results also serve as a stringent test of electronic structure calculations. In particular, for cluster molecular orbital models (i.e., models wherein the central metal atom and its nearest ligands are explicitly considered), which have been suggested as being appropriate for these

compounds. If these models are successful for the (very ionic) fluorides, they should also be applicable to other 3d metal systems.

Experimental procedures for this study are described in Section II. Results for each compound are given and discussed separately in Section III. Finally, conclusions are drawn in Section IV. Comparisons with previous photoelectron spectroscopic work on the valence-band region of 3d transition-metal compounds⁸⁻¹⁴ are made where relevant.

II. Experimental

The high-resolution XPS measurements were performed on a modified Hewlett-Parkard HP 5950A electron spectrometer.^{15,16} This spectrometer employs monochromatized AlK α X-rays ($h\nu = 1486.6$ eV) as the photon source.¹⁷ The resolution of the spectrometer is 0.55 eV.¹⁸ The system has been modified for ultra-high vacuum (UHV) studies.^{15,16}

All the specimens used in these studies were single crystals¹⁹ of the rutile (D_{4h}) structure. The samples were cleaved in situ under UHV conditions²⁰ ($5 \times 10^{-10} \leq P \leq 5 \times 10^{-9}$ Torr) in the sample preparation chamber of the spectrometer¹⁶ and immediately transported into the analyzer chamber without breaking the ultra-high vacuum. Before and after measurement of the valence band spectra, in situ analyses for carbon and oxygen contamination were performed, yielding, in most cases, undetectable amounts of these contaminants.

It is well-known that photoelectron studies of non-conducting solids is plagued with the problem of sample charging. Even though we were not interested in obtaining absolute binding energies, nonetheless, charging presented some problems due to inhomogeneous line broadening. To minimize this source of resolution degradation, the samples were bathed with low energy electrons from an electron flood gun.^{21,22} The flood gun settings were determined empirically by plotting the Fls full-width at half-maximum (FWHM) vs the flood gun voltage and current settings. Use of the flood gun resulted in narrowing the observed linewidths ~25%.

III. Results and Discussion

We will discuss each of the transition metal difluorides separately below. Table I summarizes the characteristic valence-band energies of these compounds relative to the top of the valence band.

A. MnF₂

Manganous difluoride has proven to be a very useful compound in earlier detailed studies of Mn core-level spectra which led to an experimental understanding of multiplet splitting and electron correlation in photoelectron spectra of transition metal compounds.²³ MnF₂ is the simplest transition metal fluoride except for the d⁰ case. It has a half-filled 3d shell with a $t_{2g}^3 e_g^2$ configuration. The valence region of MnF₂ (Fig. 1) exhibits two broad peaks. One extends from 0 to ~5 eV and is centered at ~3.3 eV relative to the top of the valence band, while the other is centered at about 6.6 eV and extends from ~5 to ~10 eV. With the aid of the alkali fluoride spectra,²⁴ we can safely and unambiguously assign the 6.6 eV peak to the F2p band. This follows because both the F2s-to-F2p separation and the intensity ratio are essentially constant in the various alkali fluorides. In this analysis, they matched the separation in MnF₂ of the F2s peak to the 6.6 eV peak and the relative intensity of the F2s to the 6.6 eV peak.²⁵ This deduced ordering of fluorine 2p and metal 3d agrees also with the ordering proposed by Poole et al. on the basis of the Born model analysis.¹⁴ Figure 2 shows the MnF₂ spectrum after the F2p contribution has been subtracted from the valence region. This was accomplished by:

- (1) normalizing the intensity of F2s peaks in MnF₂ to that in LiF,
- (2) aligning the F2s peak in LiF with the F2s peak in MnF₂, and

(3) taking the difference between the two spectra. The results should yield the density of 3d states plus some contribution from multielectron satellites and differences in the inelastic losses in the two compounds.

Recent UPS studies¹⁴ (Fig. 3) at 40.8 eV photon energy yield results similar to Fig. 1 for the valence region. In the UPS spectrum the 6.6 eV peak, which we have assigned to F2p region, is more intense than the 3.3 eV peak, which has been assigned to the d states. In our XPS spectrum the relative intensities of these two peaks are reversed. This confirms our interpretation, because the F2p cross-section at 40.8 eV is expected to be favored over the Mn3d cross-section when compared to these cross-sections at 1486.6 eV.

Photoionization of a 3d electron in MnF_2 results in either a 5E_g or a $^5T_{1g}$ final state with a relative intensity $(^5T_{1g})/(^5E_g) = 1.5$. We derived a value of 1.0(1) eV for the $E_g - T_{1g}$ splitting by fitting our data with this ratio. Multiple scattering X α (MSX α) calculations of Larsson and Connolly²⁶ on a MnF_6^{4-} cluster gave 1.36 eV for the splitting. Recently Viinikka and Bagus²⁷ have performed calculations on transition-metal fluoride clusters using a multiconfigurational Hartree-Fock (MCHF) treatment. This ab initio treatment yields a 1.1 eV separation between the 5E_g and $^5T_{1g}$ final states of MnF_6^{4-} , in better agreement with experiment. In the spectral region to higher binding energy from the Mn 3d and F2p peaks is some intensity which is not accounted for by either the p- or d-bands. This intensity is probably due to correlation ("shake-up") states, which are well-known in core-level spectra of transition metal compounds.²⁸⁻³³ Correlation states derived from ligand-to-metal charge transfer are predicted in this region by the

MCHF calculations (see Fig. 2).²⁷ Both calculations^{26,27} predict the p band to be too narrow. The 2p bandwidth is ~ 5.6 eV, with a full-width at half-maximum (FWHM) of ~ 3.3 eV. The MCHF calculation gives ~ 3.0 eV for the width, while the MSX α calculation predicts a width of 1.5 eV. The MCHF calculation places the p bands at ~ 4.9 eV below the E_g final state, while the MSX α calculation positions these states at ~ 3.8 eV. The experimental value is ~ 4.4 eV.

B. FeF_2

The valence-band spectrum of FeF_2 (Fig. 4) is very similar to MnF_2 except for a new, well-resolved feature at the top of the valence band. We can use the procedure applied to MnF_2 above to obtain the 3d partial density of states. This results in the partial 3d density of state spectrum of Fig. 5. Thus the region from 0 to ~ 7 eV is due to the d states and the region from ~ 7 to ~ 11.0 eV is due to the F2p states. This partitioning into p and d regions is further supported by the results of R. T. Poole et al.¹⁴ on the basis of cross-section differences between their 40.8 eV spectrum and our 1486.6 eV results. The new feature at 2.0 eV is due to final-state multiplets. Photoemission from $\text{Mn}^{+2}(3d^5)$, with a half-filled d shell, can only lead to quintet final states. In $\text{Fe}^{+2}(3d^6)$, the one extra d electron is antiparallel to the other majority-spin d electrons. Thus both quartet and sextet final states are now possible. The peak centered at 5.1 eV represents the manifold of quartet states, and the peak at 2.0 eV with about 1/5 the intensity (0.23) of the quartet states is the 6A_1 final state. A similar conclusion was drawn by Wertheim et al.³⁴ from a spectrum in which the 6A_1 state was just resolved. This points up the very

localized nature of the 3d bands. In the rare-earth metals the 4f electrons of the trivalent ion cores are well-known to be very localized, and they give similar spectra in their compounds, such as the trifluorides.³⁵ We have an analogous case in going from Gd metal with a half-filled 4f shell to Tb with one electron beyond a half-filled 4f shell. Photoemission from the Gd 4f states can only yield 7F states, while for Tb, photoionization of the 4f electrons produces a manifold of sextet states from the majority spin states plus an 8S state (minority spin state) with 1/7 the intensity of the sextet state. In FeF_2 as in MnF_2 there are also correlation states in the higher binding-energy region of the valence band. Unfortunately, there are as yet no theoretical results available for FeF_2 .

C. CoF_2

The VBDOS of CoF_2 (Fig. 6) is not as simple as those of MnF_2 or FeF_2 . However, our success in using the LiF data to decouple the F2p contribution from the 3d states in the simpler cases of MnF_2 and FeF_2 gives us confidence to apply this technique to CoF_2 . The results are shown in Fig. 7. Also shown in Fig. 7 as lines are the most intense final states predicted by the MCHF calculation of Viinikka and Bagus.²⁷ The agreement is remarkably good. The hatched area represents correlation-state structure, which is much more intense than in FeF_2 or MnF_2 . However, Co core levels of CoF_2 also exhibit intense correlation-state satellites, while in MnF_2 and FeF_2 the metal core levels have much weaker satellites.²⁹ The MCHF calculation predicts correlation states in this region, as indicated in Fig. 7. Again, the UPS¹⁴ results are in good agreement and show the expected cross-section

modulation. The p band falls about 4.7 eV below the most intense feature in the d bands. The MCHF calculation predicts this peak to fall at 7.0 eV, far too low.

D. NiF_2

The XPS VBDOS of NiF_2 , like CoF_2 is not very simple as there is a large degree of overlapping of the metal 3d bands with F2p bands. Also the NiF_2 spectrum may not be as well resolved as the above difluorides because the sample was not a single-crystal. We do get good agreement (Fig. 8) with the (polycrystalline) XPS spectrum of Wertheim et al.³⁴ Poole and co-workers¹⁴ have done UPS measurements at $h\nu = 40.8$ eV on NiF_2 . Figure 9 shows the result of subtracting out the F2p contribution. Again very reasonable agreement with MCHF calculation for the 3d states is observed. Intense correlation structure is also observed in agreement with the Ni core-level spectra in NiF_2 .²⁹ The p levels are observed to be ~ 5.2 eV below the $^4T_{1g}$ state, which compares with ~ 6.1 and ~ 3.8 eV predicted by the MCHF and MSX α calculations respectively. As in the case of MnF_2 , both calculations predict the p bands to be too narrow. The MSX α calculation yields a bandwidth of 1.8 eV, while the MCHF calculation is somewhat better with a bandwidth of ~ 3.0 eV. As in the case of MnF_2 , the MSX α calculation tends to suggest about a 1 eV splitting in the p bands, which is not observed.

E. ZnF_2

ZnF_2 was discussed earlier and we will just summarize our conclusions here.³⁶ The F2p contribution to the valence region was estimated by the above procedure of using the LiF spectrum. After subtraction,

considerable spectral intensity remained between the intense Zn3d feature and the top of the valence band. This was attributed to crystal-field split 3d levels. The interpretation is supported by the fact that if only the most intense feature is assigned to the Zn3d band, then: (1) the intensity ratio of Zn($2p_{3/2}$) to Zn(3d) is too high compared to zinc metal, and (2) the Zn($2p_{3/2}$) to Zn(3d) energy separation would be low by 1 eV compared to the metal. It is clear that the Zn3d levels are crystal-field split; however, the exact nature of this splitting is not yet known.³⁶

IV. Conclusions

For the difluorides, to a good approximation the 3d metal d bands and the F2p band are decoupled. In cases where we have MCHF calculations (MnF_2 , CoF_2 , and NiF_2), these calculations appear to give very good agreement with our derived partial d densities of states. In these calculations the 3d electrons are allowed to rearrange among themselves, all the electrons are explicitly treated, and final-state effects are treated. Both the MCHF calculation and the MSX α calculation in general do not do very well for the F2p bands. They tend especially to be too narrow. The MCHF results are much better than the MSX α . The MCHF calculation also does well in predicting the energy of the correlation states. It would be particularly useful to obtain spectra over a wide range of photon energies in the cases where there is large overlap between the d and p bands (CoF_2 and NiF_2). Such data would give a good test of the usefulness of the technique used above to obtain partial densities of states. Also angle resolved photoemission measurements would be important in further clarifying the exact nature of the partial density of d-states.³⁷

Acknowledgements

We would like to thank Drs. R. S. Feigelson, H. Rinneberg, and J. Hartman for the gifts of several single crystals. Many useful discussions with Drs. P. S. Bagus, E.-K. Viinikka, Professor L. M. Falicov, B. Koiller, and B. H. Brandow are gratefully acknowledged. We are also grateful to Professor R. C. G. Leckey for communication of the UPS results (Ref. 14) on the difluorides prior to publication.

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Table I. Characteristic features (in eV) of the transition metal difluorides (relative to top of the valence band).

	1*	2	3	4	5	6	7
MnF_2	2.2(3) [†]	3.2(3)	4.6(3)	6.6(3)	9.6(4)	--	--
FeF_2	2.0(2)	2.8(3)	5.1(2)	6.5(2)	8.0(3)	11.0(4)	14.0
CoF_2	2.5(3)	5.1(3)	7.2(3)	10.4(4)	11.2(3)	13.7(4)	--
NiF_2	4.0(3)	6.8(3)	8.4(3)	10.0(3)	11.5(3)	14.5(3)	--

* Column headings are numbers of spectral "features" in Figs. 1, 4, 6, and 8.

[†] Error in last place is given parenthetically.

Figure Captions

Fig. 1. MnF_2 : XPS F2s-valence band (VB) spectrum.

Fig. 2. MnF_2 : Partial d-density of states (DOS), obtained as discussed in text. The solid vertical bars represent calculated d-states from MCHF calculations of Ref. 27. Hatched area represents mostly shake-up intensity. The horizontal bar represents the region of predicted shake-up from Ref. 27.

Fig. 3. UPS spectra obtained at $h\nu = 40.8$ eV (after Ref. 14).

Fig. 4. FeF_2 : XPS valence-band spectrum.

Fig. 5. FeF_2 : Partial d-band density of states, resolved as described in text.

Fig. 6. CoF_2 : XPS valence-band spectrum.

Fig. 7. CoF_2 : Partial d-band density of states. See caption for Fig. 2 (the dashed vertical line indicates the sum of the adjacent lines).

Fig. 8. NiF_2 : XPS F2s-VB spectrum.

Fig. 9. NiF_2 : Partial d-band density of states. See caption for Fig. 2.

XBL7512-3937A

Fig. 1

XBL 762-2314

Fig. 2

Fig. 3

XBL766-2964

Fig. 4

XBL 762-2315

Fig. 5

XBL 757-3458a

Fig. 6

XBL 762-2317

Fig. 7

XBL 757-3409A

Fig. 8

Fig. 9

XBL 762-2316

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