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LETTERS TO THE EDITORS

Comments on Peak Assignments in the Photoelectron Spectrum of Alumina

In a recent paper Defossé *et al.* (*1*) point out the potential value of the secondary electron loss peaks, in a photoelectron spectrum, for the detection of alumina phases. We feel the spectrum they present may be misleading since the major component of the Al_{2p} satellite in their Fig. 1 is undoubtedly the Al (KLL) Auger peak, although they do not identify it as such. This Auger peak is induced by the Bremsstrahlung radiation from the X-ray source. In Fig. 1 we show in the upper curves a spectrum from alumina obtained on our V.G. ESCA 3 instrument in Al$K\alpha$ radiation. The peak observed by Defossé *et al.* is marked X. The spectrum shown in the lower set of curves is of the same sample but obtained in Mg$K\alpha$ radiation and it is clear that the peak X is no longer present. The spectra in Fig. 1 have been arranged according to a common binding energy scale to provide correlation with the same scale used by Defossé *et al.* However, it should be noted that the kinetic energies of the electrons at the peaks obtained in Al$K\alpha$ and Mg$K\alpha$ radiations, respectively, will differ by the difference in their photon energies, i.e., 233 eV. The kinetic energy scales provided show that the peak marked X, obtained in the Al$K\alpha$ radiation at a BE of $\simeq$100 eV appears in Mg$K\alpha$ radiation at the same kinetic energy, although here it is at an apparently negative binding energy. It is this behavior which identifies it as the KLL Auger line of aluminum.

An important part of the evidence presented by Defossé *et al.* in their interpretation of this peak as a discrete energy loss satellite was its correlation in appearance with the similar satellite in the O_{1s} spectrum. Curves for the oxygen 1*s* region are given in Fig. 1 and show that these peaks are not related to the peak X since they remain unchanged in the two radiations. They are similar to the broad low intensity peak seen in Mg$K\alpha$ radiation. It is this residual peak, only seen after removal of the KLL Auger peak, which corresponds to that seen in the energy loss spectra. Defossé *et al.* pointed out two consequences of the presence of the peak X. First, it can have a certain value in interpretation of the phases present in alumina/silica coprecipitates. We have not found the intensity of the Auger peak to vary markedly with phase structure as does the peak described by Defossé *et al.* However, this effect may be a combination of its decreasing intensity relative to that of the Si_{2p} line in their series of samples and a small peak shift. We have commented in a previous paper (*2*) on the useful chemical shifts of the Al (KLL) Auger peak and this could be of value in the type of work described by Defossé *et al.* For example, Fig. 2 shows the chemical shift of 6.8 eV between the Auger peak of Al^0 and Al^{3+} seen in the spectrum of partly oxidized aluminum foil in both Al$K\alpha$ and Mg$K\alpha$ radiations. While this effect can be seen in the Brems-

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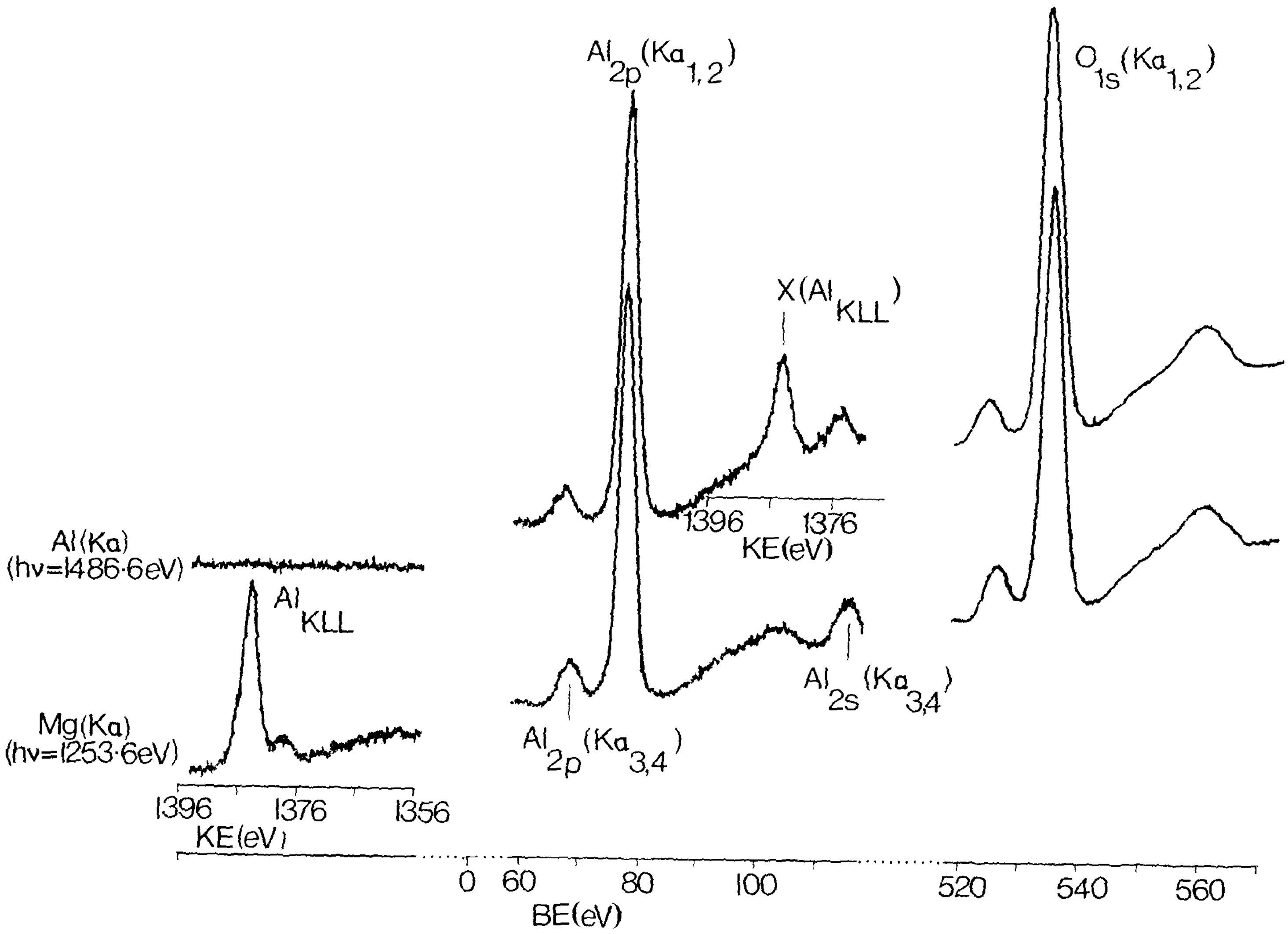

FIG. 1. Photoelectron spectra of alumina in Al$K\alpha$ radiation (upper curves) and Mg$K\alpha$ radiation (lower curves) showing the Bremsstrahlung excited Auger peak.

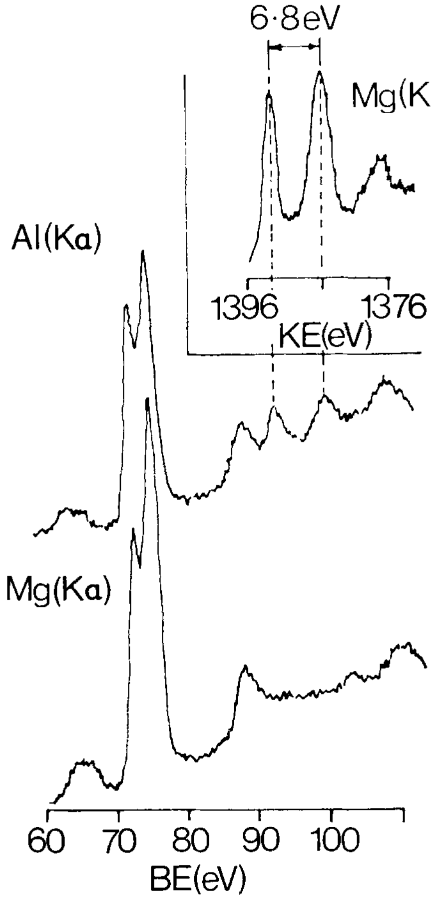

FIG. 2. Partially oxidized aluminum showing the chemical shift on the Bremsstrahlung Auger peak.

strahlung induced peak, we would recommend the use of a radiation able to excite both the Al_{1s} and Al (KLL) peaks, such as Si*K*α as described previously (*2*). We cannot agree with Defossé *et al.* that the peak intensity has any value in the identification of a particular aluminum compound.

The second consequence discussed by Defossé *et al.* is that the peak X can give an error in this quantitative interpretation of the Al/Si ratio as it falls close to the Si_{2p} peak. Since the intensity of peak X is proportional to the intensity of Bremsstrahlung radiation, which is an instrumental parameter, it has an unpredictable height relative to the Al_{2p} peak. This problem would of course be eliminated by obtaining the spectrum in either Mg*K*α or Si*K*α radiations.

REFERENCES

1. Defossé, C., Canesson, P., Rouxhet, P. G., Delmon, B., *J. Catal.* **51**, 269 (1978).
2. Castle, J. E., Hazell, L. B., Whitehead, R., *J. Electron Spectrosc. Relat. Phenom.* **9**, 247 (1976).

J. E. CASTLE
R. H. WEST

Department of Metallurgy and Materials Technology, University of Surrey, Guildford, Surrey, England

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