K emission spectra of lanthanide compounds

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Introduction

Generally, detailed analysis of x-ray emission spectra can reveal the chemical environments of the specific elements in the materials [1]. The present research aims to investigate K emission lines for lanthanide series elements, mainly in terms of changes in the line intensity ratio [2]. In view of the rather high energy (30~60 keV), however, there are still several experimental difficulties. This report describes some preliminary research for several Gd compounds.

Experimental

X-ray fluorescence (XRF) spectra were collected by an energy-dispersive spectrometer at BL14A with the storage ring operated at 2.9 GeV. The excitation energy was set at 78 keV using a Si(553) monochromator (Gd K-edge is 50.239 keV). The whole detection system is shown in Fig. 1. The fluorescent X-rays were observed by a Ge detector (Canberra GL0055PS) in the perpendicular direction, and energy resolution was estimated as 370 eV at 50 keV (Gd $K\beta_2$). Several kinds of Gd compound powder were prepared as pellets, with a diameter of 8mm and a thickness of 1mm, supported by an aluminum ring.

Results and Discussion

The XRF spectrum of Gd metal is shown in Fig 2. The accumulation time was 10^4 seconds and the dead time was kept at less than 20% under the 12 μs shaping time of the amplifier (Canberra 2021). All the emission lines were assigned as indicated in Fig. 2. The Compton peak of the 78 keV excitation line is observed around 68keV, which is in agreement with the theoretically calculated value in the direction of 90 deg. Gd $K\alpha_1$, $K\alpha_2$, and $K\beta_1$, $K\beta_2$, as well as $L\alpha_{1,2}$, and $L\beta_1$, $L\beta_2$, are observed, although the separation is not always perfect, because of the rather poor energy resolution. In addition, Ge Kα and Kβ escape peaks for some of those lines are visible. One can also see other parasitic lines originating from elements other than Gd; Ta K, Cu K, and Pb L-lines.

Although our interest is mainly in the fine structures of Kβ spectra, both $K\beta_5$ (slightly higher energy side of $K\beta_1$) and $K\beta$-$O_{2,3}$ (slightly higher energy side of $K\beta_2$) were not detected in the present study. Meanwhile, the relative intensity ratio Kβ/Kα was examined precisely for Gd compounds (metal, oxide, fluoride, nitrate, carbonate, and acetylacetonate). However, besides the chemical effects, several factors were found to be affecting the intensity ratio. Detailed analysis is under way. The authors would like to thank Prof. S. Kishimoto for his kind assistance during the experiment.

Reference

[1] L.V. Azaroff, "X-Ray Spectroscopy", McGraw-Hill, (1974).
[2] K.Sakurai and H.Eba, Nucl. Instrum. & Methods B (in press)
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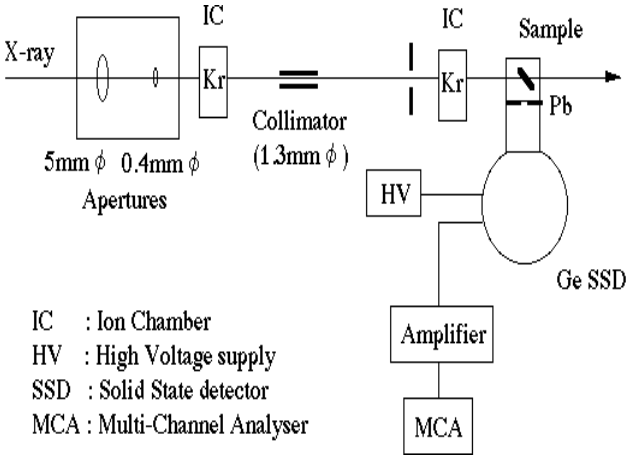

Figure 1 (up) A schematic of the high-energy XRF system.

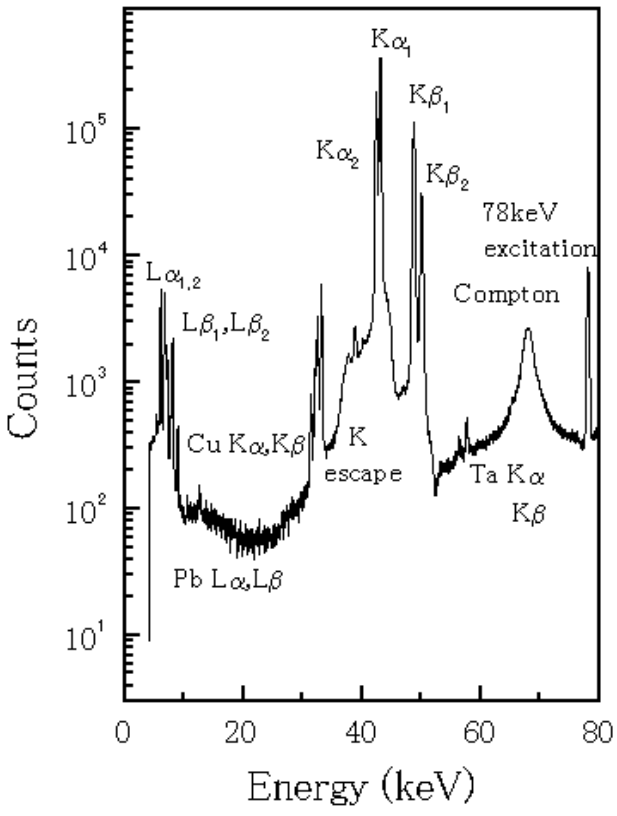

Figure 2 (right) High-energy X-ray fluorescence spectrum of Gd metal.