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Electron Probe Microanalysis of Li Kα with Newly Developed Ultra-Soft X-ray Spectrometer

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Recently, Li has been much attention to the various industrial fields as an important element. For example, $LiNbO_3$ or $LiTaO_3$ is one of the most important compounds because of its various optical characteristic and ferroelectrics. It is well known that $LiCO_3$ shows the drastic effect as a anti-depressant drag. Thus, it can be said that the necessity of the chemical state analysis for Li has been increased so much. However, Li is also well known as the one of the most difficult elements for analysis. This group has already developed the general-purpose electron beam excitation ultra-soft X-ray analyzer with new designed high-resolution flat-field VLS(Varied-Line-Spacing) grating spectrometer. The schematic view is shown in Fig.1. In this report, the preliminary results of the measurement of Li Kα for metallic Li and some Li compounds are presented.

The metallic Li is so active material as known well, and the sample was cut off in order to expose the fresh plane in the aire just before setting. No signal originated from the Li atom was detected just after the sample introduction due to the oxidation by the air. In measurement of Auger spectrum simultaneously, the Auger signal of Li was observed. Thus, This speculation might be correct, and the reason that no X-ray signal of Li was detected is now investigating.

After 30 minutes sputtering with 4keV Ar^+, the signal of Li Kα were detected as shown in Fig.2. In this figure, the spectra of LiF with Ar^+ sputtering and of metallic Li reported by Sagawa et al.[1]. The examples of “matrix” and “Top of needle” are shown in the photograph (Fig.3). This was taken in just observing the signal of Li after Ar^+ sputtering. The Auger spectra obtained simultaneously was also shown in Fig.4.

The “Matrix” of the Auger spectrum in Fig.4, the amounts of Oxygen and nitrogen are so small, and the structure of Li LVV is also simple. It seems that Li Kα from “Matrix” shows metallic Li. The shape of this spectrum agrees well with the report by Sagawa et al.[1]
On the other hand, Th Auger spectrum from “Top of needle” shows some extent existence of oxygen and nitrogen at that position. And, this Auger spectrum also shows that Li partially makes the compounds because the structure of Li LVV is more complicated than that of the “Matrix”. However, as shown in Fig.2, the energy position and profile of Li Kα at this part is almost same as the other spectra. One of possible explanation of this observation is that the yield of Li Kα of any Li compound is extremely lower than metallic Li. In Fig.5, the Li Kα spectra of LiF, $LiNbO_3$ and $LiTaO_3$ are shown as the preliminary measurement results. The intensity of spectra of $LiNbO_3$ or $LiTaO_3$ are so weak, and this observation seems to support this speculation that the yield of Li Kα of Li compound is so low. The discussion about Li Kα of LiF will be presented at the presentation.

[1] T.Sagawa, Y.Iguchie, M.Sasanuma et al., J.Phys.Soc.Jpn 21 (1966) 2587

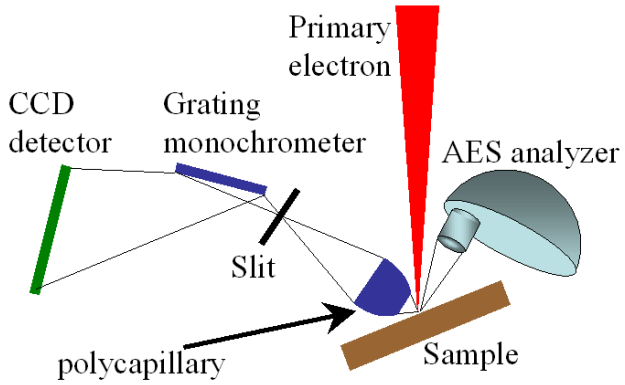

Fig.1. The schematic view of new ultra-soft X-ray spectrometer

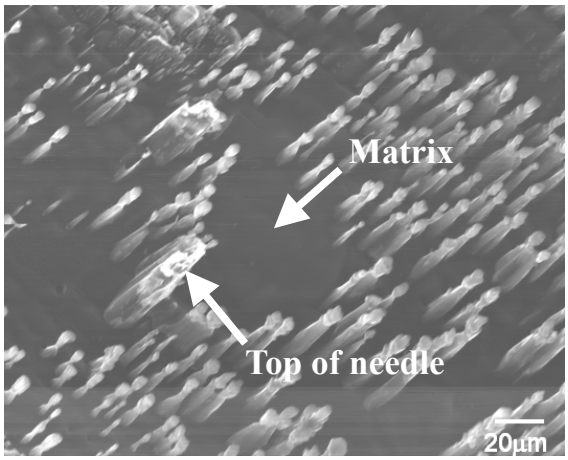

Fig.2. Observation of surface of metallic Li by SEM after Ar ion sputter at 4keV 30min.

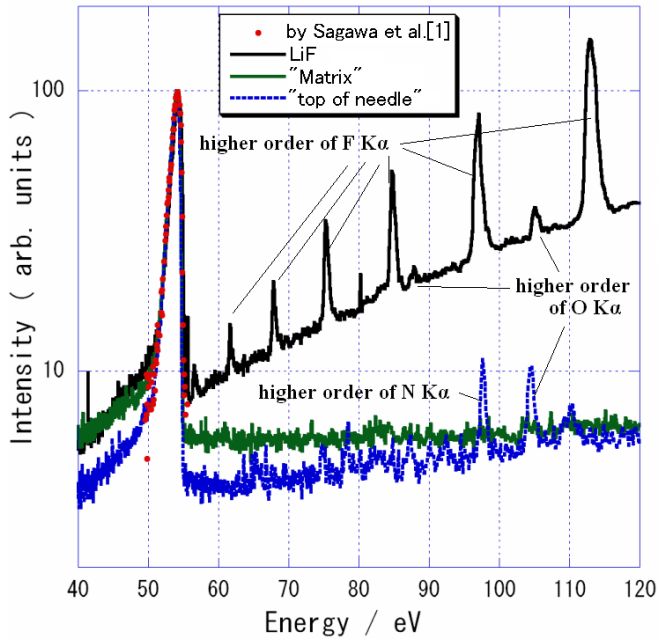

Fig.3. The comparison between various Li Kα spectra.

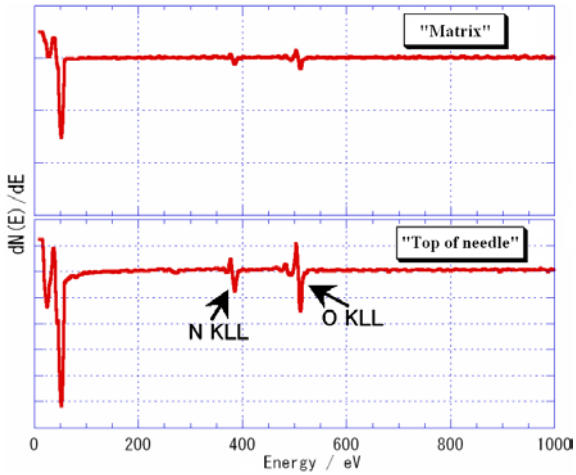

Fig.4. Li Auger spectra after Ar ion sputtering.

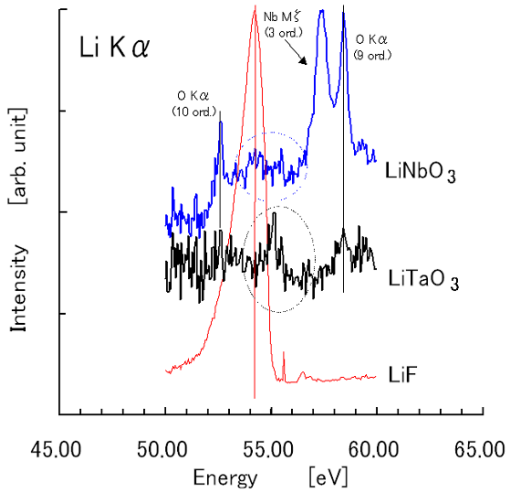

Fig.5. Li Kα spectra of LiF, $LiNbO_3$ and $LiTaO_3$.

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