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Trials to grow single crystals of the filled skutterudite compounds at high pressures

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A class of compounds with the filled skutterudite structure, RT_4X_{12} (R = rare earth, T = Fe, Ru and Os, X = P, As and Sb), has recently attracted much attention because of its variety of physical properties. For some combination of R , T and X , high quality single crystals have been grown by the flux method which has played an essential role in investigating the exotic physical properties of these materials so far. On the other hand, there still exist a large number of unsynthesized compounds, mainly RT_4As_{12} system. For such compounds, high-pressure synthesis is thought to be a powerful technique.

We are now trying to synthesize single crystal of $PrFe_4Sb_{12}$ which could not be synthesized by the flux method. The constituent elements of Pr, Fe and Sb were placed in BN crucible with a stoichiometric ratio and compressed to 4.0 GPa at room temperature. The sample was heated to 950 °C and kept for 30 minutes. Soon after, the sample was furnace cooled to 700 °C and cooled to 600 °C for 120 minutes. Figure 1 shows photograph of a sample synthesized by this condition and Fig. 2 shows a powder X-ray diffract spectra of this sample. The phase of $PrFe_4Sb_{12}$ is confirmed, although the impurity phases ($FeSb_2$, Sb) coexist. In subsequent experiments, we have confirmed the impurity phases to be reduced by changing cooling condition. $PrFe_4Sb_{12}$ single crystal has not been grown yet, we expect that it can be grown by changing cooling condition. Additionally, we are trying to synthesize another single crystals of filled skutterudite compounds $LaFe_4Sb_{12}$ and $PrCo_4Sb_{12}$.

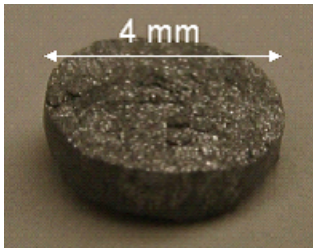


Figure 1: Photograph of a sample

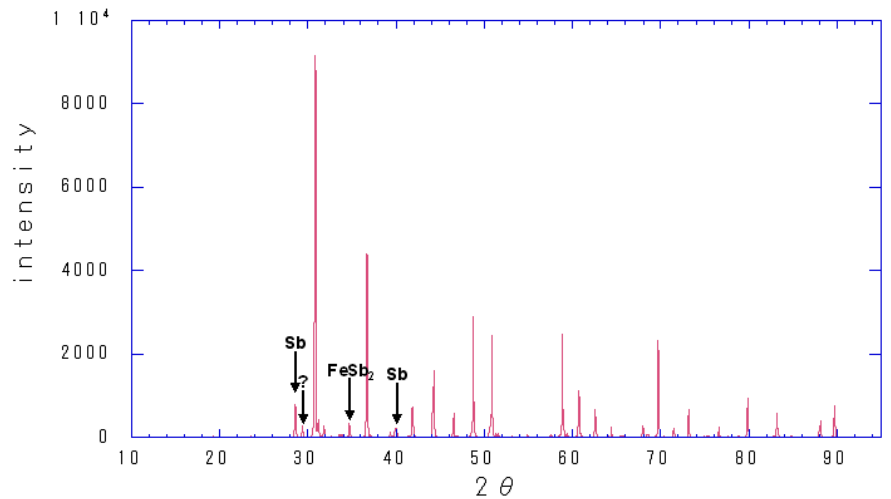


Figure 2: Powder X-ray diffract spectra are taken with Cu K_{α} radiation. The impurities peaks of $FeSb_2$ and Sb are indicated by arrows, the other peaks are $PrFe_4Sb_{12}$