# §25. Feasibility Study on the Anion Doped Oxide - Organic Nano Composite for Energy Utilization

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The final purpose of this study is partial fluorinated tin oxide (SnO2:F) and synthesis of nanocomposite consisting of SnO2:F and carbon. As the purpose, a mecahnochemical process (MCP) and a microwave irradiation one (MWR) were used. The behavior of physicochemical changed by MCP, MWR was investigated to measure by X-ray diffraction (XRD) and X-rays photoelectron spectroscopy (XPS).

The samples were used PolyVinylidene DiFluoride (PVdF), Arkema Inc. and SnO2 (Aldrich, -325mesh). MCP were performed by a planetary mill (Fritsch, Pulverisette 7) with 18 balls of 10 mm diameter made from cobalt bonded tungsten carbide (WC). A mixture comprising 3 (g) SnO2 and 0.3 (g) PVdF was subjected to milling at 800 (rpm) for periods up to an hour. The milling was carried out in a tightly sealed vial without evacuating or controlling the atmosphere.

The magnetic or electric fields of microwave can be separated on positions in the TE103 single mode cavity with the cross-section of 54.61 mm x 109.22 mm at the frequency 2.45 GHz. The infrared pyrometer, FTZ-R220, Japan Sensor CO., LTD. measured the temperature of the sample. Heating processs were carried out microwave electric field heating (E-mode), magnetic field heating (M-mode) with microwave input power (10W - 200W), and infrared heating mode (IR-mode).

Table 1 shows the sample name and sample conditions. Figure 1 sows to compar XRD profiles on the sample heated for an hour at 600 $^{O}$C in vaccume and air. The peaks of SnO2 lines were only obserbed in the sample (SV10-IR600A) heated in air at 600 $^{O}$C. On the other hand, the mixed peaks of Sn and SnO2 line in the sample heated in vacuum. In addition, the (111) rutile diffraction peaks of sample (SV00-M600V) without MCP clearly shows a shift toward higher scattering angles compaered the sample (SV10-M600V) with MCP as showing in Figure 2, implying a decrease in these interplanar distances by ca. 0.3 %.

For estimation with the he chemical states of fluorine, the samples measured by XPS, 1600-CNIS (ULVAC Inc.), under vacuum condition. The XPS spectrum is peaked at 687.8 eV and 685.2 eV in respects of Sn-F and F1s, respectively [1]. Figure 3 shows the XPS spectra with SV10, SV10-M400A and SV10-E600A. The Sn-F peaks was relatively increased with F1s peaks in SV10-M400A. And XPS intensity of SV10-E600A was decreased for dissipating the fluorine.

As a experimental results, it suggest that the MWR process was effectively for the removing the remained PvdF and increasing the Sn-F bonding under 400 $^{O}$C.

1). M. Senna et al, Solis State Sci.30 (2014), 36-43

Table 1 Sample name and preparative condition

| Starting mixture | Sample ID | Preparative condition | | |
|---|---|---|---|---|
| SV00[a)] | SV00 | As-mixed (without MW) | | |
| | SV00-M600-A | MW-Mag | 600oC, 1h | Air |
| | SV00-M150W-A | MW-Mag | 150W,1h (max 700oC) | Air |
| | SV00-M600-V | MW-Mag | 600oC, 1h | Vac |
| SV10[b)] | SV10 | As-milled (without MW) | | |
| | SV10-E600A | MW-Elec | 600oC, 1h | Air |
| | SV10-M400A | MW-Mag | 400oC, 1h | Air |
| | SV10-M600V | MW-Mag | 600oC, 1h | Vac |
| | SV-IR600A | IR | 600oC, 1h | Air |

a) $SnO_2$+PVdF, as-mixed
b) SV00, co-milled for 1h

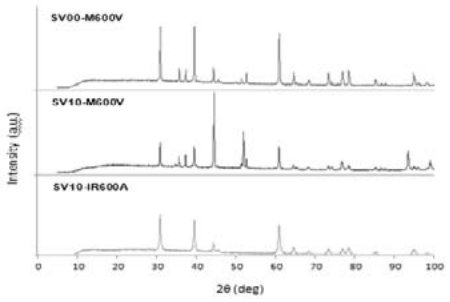


Fig. 1 XRD profiles of the sample heated for 1 hour at 600 degrees Celsius in vaccume and air

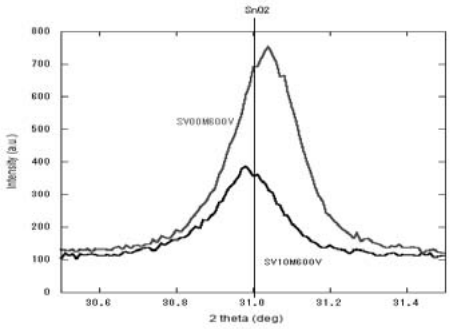


Fig. 2 (111) peaks of XRD compared the samples heated for 1 hour at 600 degrees Celsius in vaccume with and without the MCP

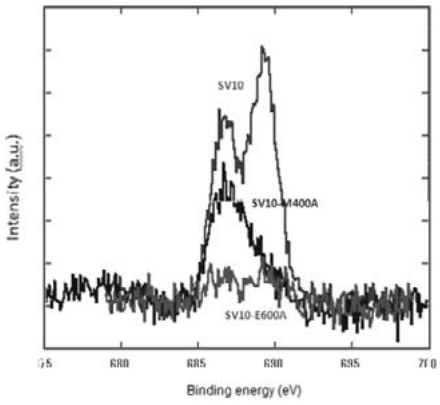


Fig.3 XPS spectra with SV10, SV10-M400A and SV10-E600A