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# DEVELOPMENT OF COAXIAL TYPE FLOW MICROWAVE REACTOR

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We have developed various type microwave chemical reactors with solid state microwave generators[1),2)]. In this report, flow microwave chemical reactors with coaxial cavities with 2.45GHz and 5.8 GHz microwave is described.

1. Design of Coaxial Cavity Chamber for 2.45GHz microwave

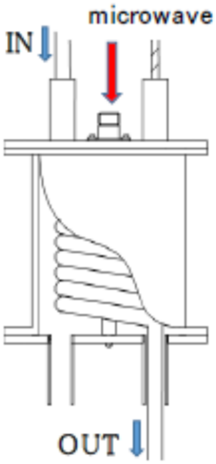



**Fig. 1.** Structure of coaxial cavity chamber for microwave chemical reaction system.

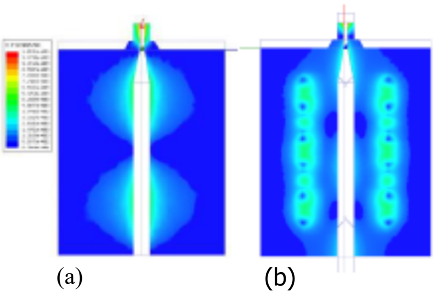



**Fig. 2.** Field distributions in the coaxial cavity chamber at 2.45 GHz. (a) Electrical field distribution (without glass tube). (b) Magnetic field distribution (with ethylene glycol flowing glass tube).

Figure 1 shows a structure of the coaxial cavity chamber for the 2.45 GHz microwave chemical system. It consists of a cylindrical cavity of 100 mm inside diameter with a metal rod at the center, containing a spiral glass tube to flow solvents. When the input port of the microwave is placed at the end of the metal rod, TEM mode is excited in the cylindrical chamber due to the presence of the metal rod.
Figure 2 (a) displays an electrical field distribution in the coaxial cavity chamber simulated at 2.45 GHz. The two maximum positions of the electric field are found. In this work, the height of the cavity is selected such that the standing wave has two maximum positions in the electric field. In this case, 120 mm is used. In this case N-type connector is used.
To achieve matched state and avoid excess heating of the input connector, a conical aperture is introduced between the N-type connector and the coaxial cavity. In addition, the end of the center rod is tapered. In order to obtain a matched state, some external matching networks should be connected. In this work, a slug tuner is used.

Figure 2 (b) shows a magnetic field distribution in the coaxial cavity chamber with the glass tube simulated at 2.45 GHz. As a solvent in the glass tube, ethylene glycol is assumed. It is found that the magnetic field is uniform in the glass tube. So, the equal microwave heating is expected in the coaxial cavity chamber. This configuration was confirmed suitable for rapid and continuous microwave syntheses of various functional metal complexes. Experimental results are presented for synthesizing Ir(III) complexes for OLED dopants and Ru(II) complexes for various sensors.

2. Microwave syntheses of Ir(III) complex and Ru(II) complex with a flow microwave reactor with a coaxial cavity.

Ir(III) and Ru(II) complexes are important functional materials as phosphor illuminants or phosphor sensors. As for syntheses of these metal complexes, it is a hard process to dissociate M-Cl bond of $MCl_3$ to create new bonds with organic ligands; accordingly effective new synthetic methods of these complexes are strongly required. Conventional synsesis of Ir(III) and Ru(II) complexes are tedious, time-consuming, andlow yeild of products.

Using microwave reaction, these difficulties are resolved; rapid and high yield synthetic methods have been reported .[3),4)]

Microwave syntheses of these metal complexes are performed with the newly developed coaxial flow microwave reactor as shown in Fg.3.

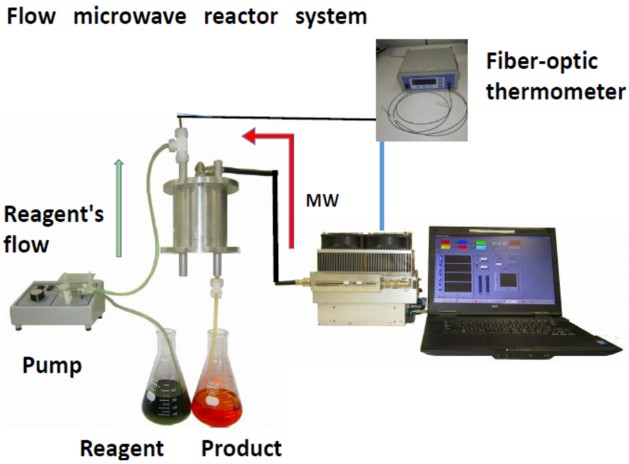



**Fig.3** A coaxial flow microwave reactior system

Firpic, [2-(4,6-difluorophenyl)pyridinato-$C^2$,*N*] picolinato)iridium(III) which is one of blue OLED dopants, was synthsized with the coaxial flow microwave reactor system. The reaction porcess is compared with the conventional synthesis in the table.

**Table.** Comparison of classical and flow microwave reactor syntheses.

| Synthetic procedure | Reaction time | Power consumption | Purification procedure | Yield |
|---|---|---|---|---|
| Classical synthesis | 18.5 hours | 2800 WH | Column Separation<br>Long time, Much Solvents | 53% |
| Flow microwav e method | 0.75 hours | 75 WH | Recrystallization<br>Short time,<br>Small amount Solvents | >75% |

3. Property of 5.8GHz flow microwave reactor

In the similar manner as the coaxial reaction chamber of 2.45GHz, a coaxial reaction chamber for 5.8GHz IMS band is designed with 51mm diameter and 50mm height. The electric field distributions in the chamber and the temperature profiles of solvent are simulated using the commercial simulator (COMSOL Multiphysics) for 5.8 GHz, 5W microwave input.

The structure of the coaxial line type reaction chamber is shown in Fig.3. Metallic rods of diameter $c_d$ and height $m_h$ are installed as a center conductor in the cylindrical metal cavity (diameter $m_d$, height $m_h$). The tip of the center conductor $c_l$ has the form of a truncated cone of diameters $u_{d1}$ (upper base) and $c_d$ (lower base). A lid of thickness $l_h$ is attached to the top of the cylindrical cavity. A truncated conical hole with diameters $u_{d2}$ (upper base) and diameter $b_{d2}$ (lower base) is used for the coaxial input port of microwave. Selecting the size of the coaxial cavity appropriately, the places where the incident and reflected microwaves are added are generated. By arranging a flow channel (PTFE) which has the outer diameter $o_d$ (and the inner diameter $i_d$) in a spiral shape with the interval $p_i$
at the distance $r$ from the center conductor, it is possible to raise the temperature of the solvent in the channel in the chamber.

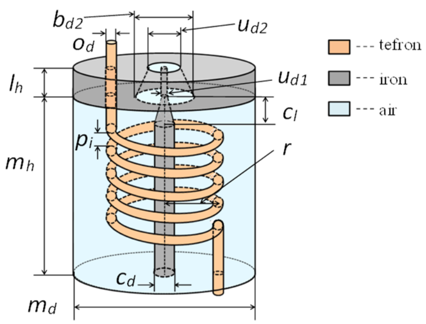



**Fig.3** Structure of reaction chamber.

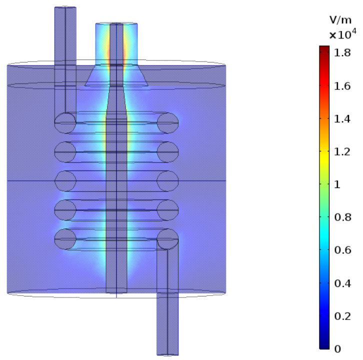



**Fig .4** Simulated electric field distribution.

For the input port of the microwave, an N type coaxial connector is assumed, and the upper base $u_{d2}$ = 10 mm of the truncated cone shape of the lid and the diameter $u_{d1}$ = 3.1 mm of the upper base of the truncated cone part are determined. The dimensions are adjusted so as to realize the low microwave reflection characteristics. As a result, the lower base $b_{d2}$ = 16 mm of the truncated cone shape of the lid, the thickness $l_h$ = 5 mm, the diameter of the central axis $c_d$ = 5 mm, the length from the tip $c_l$ = 8 mm are determined. The electric field distribution obtained by the simulation is shown in Fig.4.

4. Numerical Simulation of Temperature Profiles

The temperature of water for 5.8GHz, 5W microwave input is examined using the COMSOL. The flow channel installed inside the cavity is assumed to be made of PTFE. However, in this simulation, the thickness PTFE is ignored, that is, the water is arranged in the spiral form at the distance $r$ = 12 mm from the center conductor and the interval $p_i$ = 2 mm, with the outer diameter $o_d$ = 5 mm. The temperature distribution after 300 seconds is shown in Fig.3. The temperature-time profiles at each point P1, P2, P3, P4, and P5 in Fig.3 is shown in Fig.4. In the simulation, the complex relative permittivity of water, 72 - j 20, thermal conductivity, 0.615W/(mK), specific heat capacity 4.2 kJ/(kg K), initial temperature 20 ° C is assumed. From Figs 5 and 6, it is found that the temperature of water rises over 90 ° C at P3 point.

The temperature-time profiles of water were simulated. From the results, it was confirmed that the temperature rises to 95 ° C in 300 seconds, when the microwave power of 5W is input. This structure is smaller than that of 2.45GHz and can be used in less space.

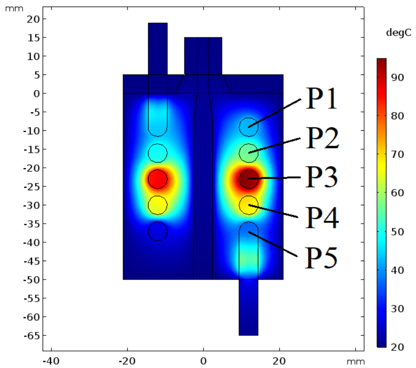



**Fig. 5** Simulated temperature distribution.

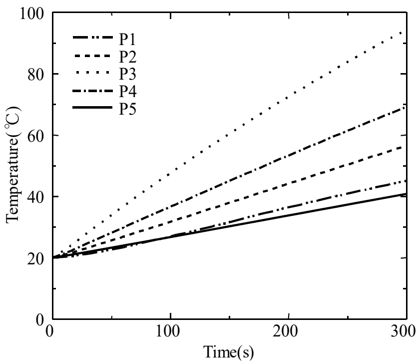
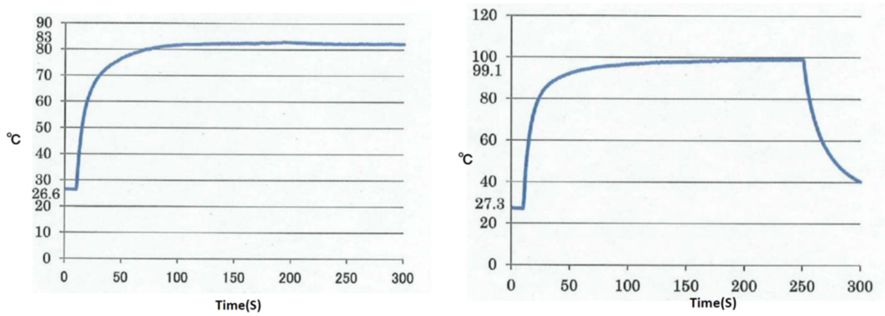



**Fig.6** Temperature – time profiles of water under microwave irradiation (5.8GHz, 5W).

Temperature profiles for experiments are shown in Figure 7 using ethyleneglycol and ethyl alcohol. Thus, the effect of microwave at 5.8GHz, with the higher frequency, the thermal effect became larger with hiher effciency.




(a) Ethyleneglycol (b) Ethyl alcohol

**Fig.7.** Temperature Profile for (a)Ethyleneglycol and (b)Ethyl alcohol

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