中性子線CTによる超臨界水熱合成 反応器内混合状態のin-situ観察

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Hydrothermal synthesis

Hydrothermal synthesis of metal oxide nanoparticles

 $Zn^{2+} + 2H_2O \rightarrow Zn(OH)_2 + 2 H^+$ $Zn(OH)_2 \rightarrow ZnO + H_2O$

Plug-flow reactor



Effects of mixing

ZnO



500nm



T.Adschiri, et al., in Materials Chemistry in Supercritical Fluids, Research Signpost, 79–97 (2005)

Mixing of supercritical water and reactants affects the products

Visualization techniques

View cell

Fluid dynamics simulation





Design of mixer

Aizawa, et al., J. Supercrit. Fluids **43**, 222 (2007).

Wakashima, et al., J. Chem. Eng. Jpn. **40**, 622 (2007).

Visualization of real apparatus

Previous studies revealed

- Buoyancy force
- Density difference
- Natural convection
- Cascade down
- Reactor design
- Rate of heating
- Mixing time

Shape of our mixing components



How the streams mix in the real apparatus ?
Neutron radiography

Why neutron radiography ?

Plug-flow reactor 1.0 Heater Heater Cooler 0.8 Density of water (g/cm³) 0.6 Pressure valve 0.4 0.2 Metal oxide Metal ion Water nanoparticles 0.0 solution 100 200 300 400 500 Temperature (°C) 300~400°C, 25 MPa

Density of water @ 25 MPa

Density of water drops at the mixing point.

Previous studies





Averaged images were obtained. CT experiments

How to obtain CT images ?





Experimental condition



Outer diameter: 1/8 inch Inner diameter: 2.3 mm P = 25 MPa

Imaging area: 65×65 mm²

60 s for 1 image 200 images for one condition

2012/10/23~25 @ B4, 5 MW

Heated water from top



Heated water from side















Summary

中性子線CT測定を行い、超臨界水熱合成反応器内の 流動・混合状態の3次元測定の可能性を示した。

今後の展開

- 流体シミュレーション計算の妥当性検証
- より解像度の高い観察
- 実際の生成物との比較

謝 辞 京都大学原子炉実験所 川端先生、齊藤先生、伊藤先生 神戸大学 竹中研のスタッフ及び学生の方々