

# Controllable Solute-Diffusion In-Gel Growth of BCHT: An Effective Approach Towards Large Functional Material Single Crystal Synthesis

Ibrahim Hany <sup>a</sup>, Bryant Kanies <sup>a</sup>, and Ge Yang <sup>\*a</sup>

<sup>a</sup> Department of Nuclear Engineering, North Carolina State University, Raleigh, NC 27607, USA.

## Experimental Methods

### Gel Formation & Growth Trial

The sodium silicate gel was formed in a modified U-Tube. Sodium silicate gel is formed by combining waterglass solution (WGS) with hydrochloric acid (HCl). To form WGS, sodium silicate solution is diluted with H<sub>2</sub>O. The volume of the sodium silicate solution is calculated as follows:

$$V_{sss} = V_d \frac{[1 - \rho_d]}{[1 - \rho_{sss}]} \#(1)$$

Where  $V_d$  is the volume of the WGS needed,  $V_{sss}$  is the volume of the sodium silicate solution,  $\rho_d$  is the desired density, and  $\rho_{sss}$  is the density of the sodium silicate solution. In this study, a sodium silicate solution density of 1.28 and a desired density of 1.06 were used. To form the silicate gel, the WGS was added to the HCl while stirring. After a 48-hour period of gelation, the precursor solutions were placed on top of the gel and allowed to diffuse over a two-week period. The first precursor solution was formed by dissolving 10 g of Bi<sub>2</sub>O<sub>3</sub> in 100 ml of 7N HCl. This was then placed in the arms of the U-Tube on top of the gel. The second reactant was made by dissolving 9.33 g of thiourea in 93.33 ml of H<sub>2</sub>O. This was placed in the center portion of the U-Tube above the gel.

## Results & Discussion:

### Gel Crystal Growth

The double diffusion gel growth technique is remarkably efficient. Figure S1 displays the crystals harvested from one growth trial. On the left, several single crystal samples can be observed ranging in size from <5 mm to nearly 1 cm. On the right, polycrystalline samples can be observed ranging from only a few mm to nearly 2 cm. This demonstrates again the great potential of this gel growth method.

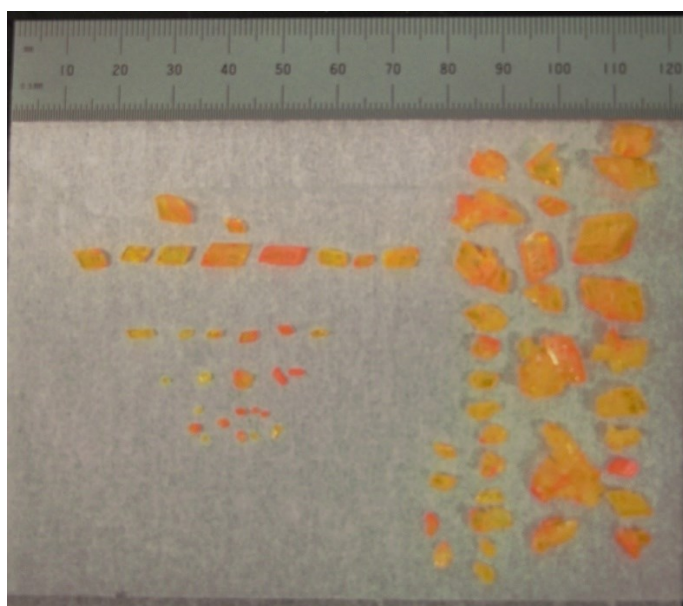


Figure S1: Crystal harvest of a single growth run. The left half of the image shows several single crystal samples, while the right-half displays polycrystalline samples.



Figure S2: Close up of several single crystal samples following harvest.