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CRYSTALLIZATION OF 1, 2, 4, 5-TETRABROMOBENZENE UNDER NANOSCALE CONFINEMENT

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Thermosalient (TS) crystals are an emerging class of organic materials that undergo macroscopic motion when taken over their phase transition. The visually observable motion occurs over distances that are several times greater compared to the size of the crystals. Such efficient conversion of heat into mechanical work favors these crystals for application as thermomechanical actuators. Recent reports have shown that crystalline organic compounds exhibit unusual polymorphism and preferred orientation when confined within nanospaces. Inspired by such behavior and in an attempt to observe the TS effect at the nanoscale, 1, 2, 4, 5-tetrabromobenzene (TBB) was selected as a model material that undergoes TS phase transition from β to γ phase at about 40°C. To that end, TBB was crystallized within the 20-200 nm-wide channels of anodic aluminum oxide (AAO) templates, and its structure was studied using two-dimensional x-ray microdiffraction (2D-μXRD) and powder x-ray diffraction (PXRD) analyses. Whereas bulk TBB crystallizes in the β phase at room temperature, the analysis revealed that when confined within the anodized aluminum oxide (AAO) nanopores, TBB crystallizes in the metastable γ phase with preferred orientation at room temperature. Figure 1a shows the 100 nm pores of AAO templates filled with TBB, whereas Figure 1b shows TBB crystals released after dissolving the AAO templates. The 2D-μXRD pattern of the TBB filled AAO template in Figure 1c shows discrete spots, revealing preferred growth of the TBB nanocrystals. The PXRD pattern of TBB-filled AAO template, shown in Figure 1d, is identical to that of bulk γ phase TBB, confirming that TBB grows in the γ phase within the nanopores. We also found that these TBB nanocrystals remain indefinitely stable in the γ phase from cryogenic temperatures up to nearly 80°C, where they sublime. These findings uncover fundamental differences between nanosized organic TS crystals and their bulk counterparts.

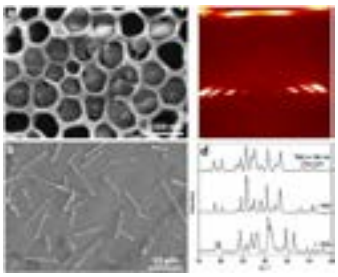

Figure 1: (a,b) SEM images of TBB-filled AAO template (a) and TBB nanocrystals obtained after dissolving the AAO template (b)&(c) 2D-μXRD pattern of TBB filled AAO template. (d) PXRD pattern of crushed TBB-filled AAO template shown together with PXRD pattern of bulk β and γ phase of TBB crystals.

Recent Publications

1. **N K Nath, M K Panda, S C Sahoo and P Naumov (2014) Thermally induced and photoinduced mechanical effects in molecular single crystals - a revival. CrystEngComm 16(10):1850-1858.**
2. **P Naumov, S Chizhik, M K Panda, N K Nath and E Boldyreva (2015) Mechanically responsive molecular crystals. Chemical Reviews 115(22):12440-12490.**
3. **C L Jackson and G B McKenna (1996) Vitrification and crystallization of organic liquids confined to nanoscale pores. Chemistry of Materials 8(8):2128-2137.**
4. **H F Lieberman, R J Davey and D M T Newsham (2000) Br···Br and Br···H interactions in action: polymorphism, hopping, and twinning in 1,2,4,5-tetrabromobenzene. Chemistry of Materials 12(2):490–494.**
5. **Khalil C, T Hu and P Naumov (2018) Nanoscale crystallization and thermal behaviour of 1,2,4,5-tetrabromobenzene. CrystEngComm 20:636-642.**

Biography

Abdullah Khalil is currently a Postdoctoral Associate at New York University, Abu Dhabi since July 2016. He obtained his PhD degree in May 2016 while working at the Advanced Fibers and Biofuel Laboratory at Masdar Institute of Science & Technology in the United Arab Emirates where he investigated the microstructure evolution and applications of electrospun metal oxide nanofibers. His research interests include synthesis of various organic and inorganic nanostructures with controlled microstructure and chemistry for functional applications. He has also contributed to studies of the microstructure and chemistry of nanomaterials by using microscopic and spectroscopic techniques. He is also experienced with using transmission electron microscopy in which he uses to study microstructure evolution in nanostructured materials.

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