Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Metal-coated thermosalient crystals as electrical fuses

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Materials and methods

Materials. 1,2,4,5-tetrabromobenzene powder (TBB, 97%, Sigma Aldrich) was recrystallized from toluene (HPLC grade, VWR Chemicals). Silver nitrate (Analytical reagent grade, Fisher Scientific), ammonia solution (VWR Chemicals), sodium hydroxide (98%, Sigma Aldrich) and dextrose (Sigma Aldrich) were used as reactants for coating of the TBB crystals. The water was purified through a Millipore system (Milli-Q Integral 10).

Synthesis of TBB crystals. Saturated solution of TBB was prepared in toluene at room temperature and then filtered to remove any dust particles. The filtrate was transferred to 2 mL-amber vials which were covered with aluminum foil. The solutions were maintained at 50 °C for 10–12 days in an incubator which ensured very small temperature variation, and this resulted in formation of long prismatic crystals with nearly square or rectangular cross-section. Most crystals were twinned, as concluded by observation with optical microscope. The cross-sectional dimensions of the crystals varied between 100 μm and 1.5 mm, and the length varied between 1–7 mm. For the fuse link experiments, crystals with an average cross-sectional dimensions of 1 mm and length 3–7 mm were manually selected and coated with silver. Crystallization attempts at room temperature did not afford crystals of sufficient size, and the maximum crystal length was found to be under 1 mm. To obtain crystals of sufficient size required for incorporation into the electrical circuit, the crystals were grown in the γ form at 50 °C, following a previously published procedure (reference 16 in the main text). After the crystallization, the crystals were extracted at room temperature. Minor cracking and splitting of the crystals was occasionally observed which is due their transition to the β form, however, this did not have a significant effect on the overall shape and size of the crystals.

Coating TBB crystals with silver. The TBB crystals were coated with silver by using the well-known Tollens' reaction where elemental silver is deposited by reduction of silver nitrate with an aldehyde. In a typical reaction, 5 mL of 0.1 mol L^{-1} aqueous silver nitrate solution was taken in a 10 mL flask, and a few drops of 15 mol L^{-1} aqueous ammonia and then 1.5 mL of 0.8 mol L^{-1} aqueous sodium hydroxide solution were added. Finally, 1 mL of 0.25 mol L^{-1} aqueous dextrose solution was added as reducing agent. As the reaction started, crystals of TBB were dropped in the flask which was constantly shaken until a shiny mirror surface formed on the walls of the flask. The crystals, which had silver mirror deposited on their surface, were then removed from the flask and washed several times with deionized water.

Characterization. The surface and cross-sectional morphology of the Ag/TBB crystals were analyzed with FEI Scios scanning electron microscope (SEM), by using the beam accelerating voltage and current of 5 kV and 25 pA, respectively. The powder X-ray diffraction (PXRD) analysis of the crystals was performed at room temperature with Empyrean X-ray diffractometer in the θ range of 5–50° using CuK_α (0.154 nm) radiation.

Thermosalient response of Ag/TBB hybrid crystals. The thermosalient response of Ag/TBB crystals was compared with that of pure TBB crystals by simultaneously heating one of each crystals on a glass placed on a hot plate and observed with optical microscope (Nikon SMZ18). The response was recorded at 1000 frames/second using high-speed camera (HotShot 1280CC, Nac Image Tech) attached to the optical microscope.

Testing of Ag/TBB crystals as electrical fuse links. To test the performance of the hybrid Ag/TBB crystals as electrical fuse links, a simple circuit was fabricated with a copper trace thickness of 18 μm. As shown in Figure S5, the circuit consists of four components: voltage supply, resistor, light-emitting diode and the Ag/TBB crystal as fuse link. The ends of the Ag/TBB crystal were connected to copper pads by using tiny droplet of water-based silver suspension. The current-voltage characteristics of individual crystals were measured with digital data logger (LabQuest2, Vernier instruments) equipped with current and voltage sensors. In this case, the LED was replaced with an ammeter (LabQuest2 current sensor). The temperature profile of the coated crystals during the experiments was recorded by using infrared thermographic camera (ImageIR, InfraTec Inc.).

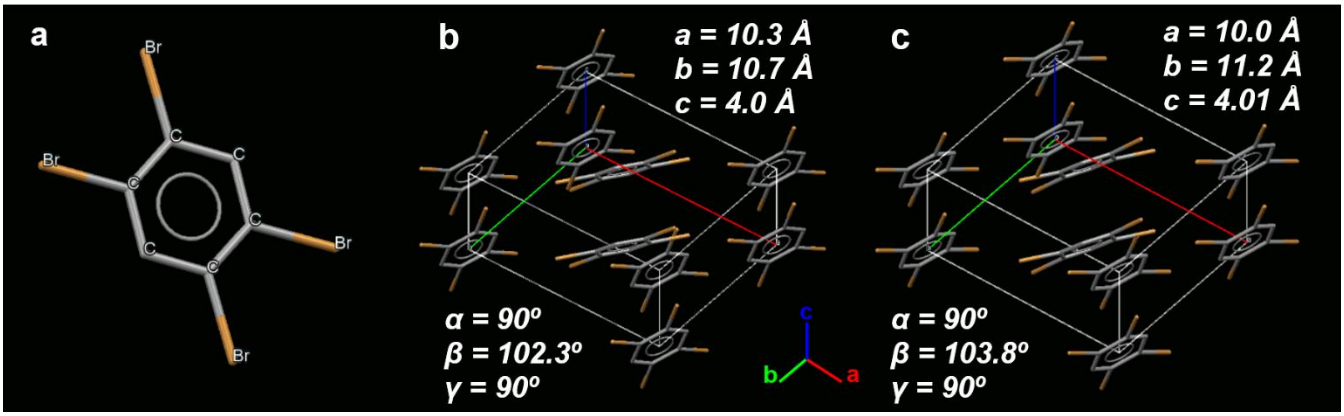

Figure S1. Molecular and crystal structure of TBB. (a) Molecular structure of TBB. (b,c) Packing in the crystal structures of the β phase (b) and the γ phase (c) of TBB.

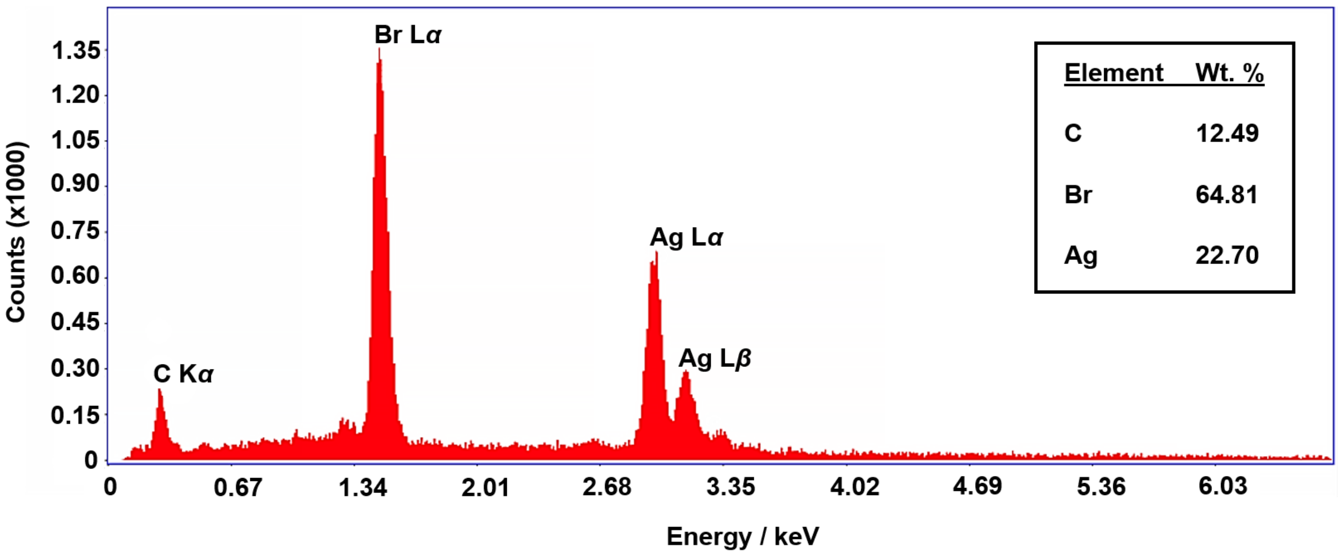

Figure S2. EDS spectrum recorded from the surface of Ag/TBB crystal. The inset shows the approximate elemental composition of the surface (in weight percent).

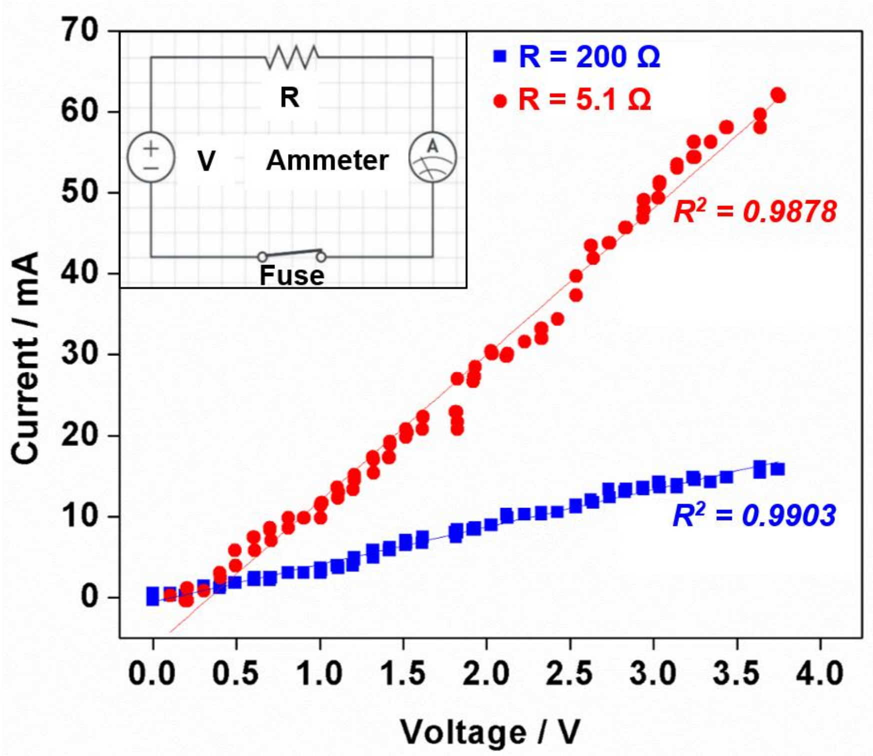

Figure S3. I–V characteristics of Ag/TBB crystal („Fuse“ in the inset) recorded under two different values of the resistance („R“ in the inset). The inset shows schematic of the circuit used to record the curves.

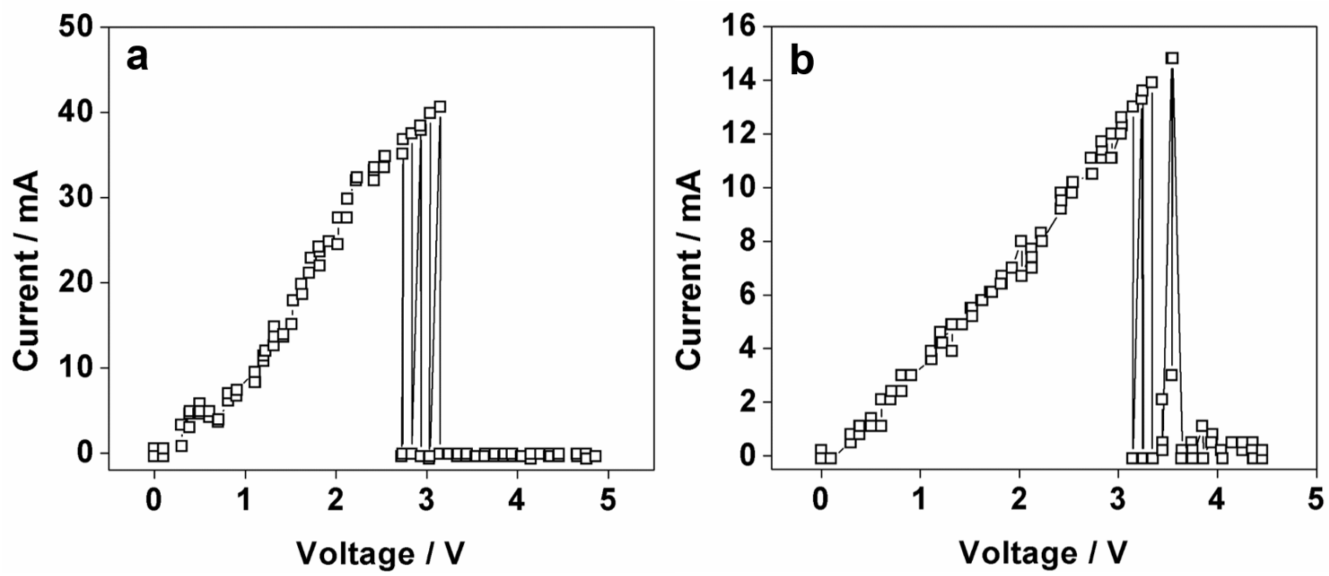

Figure S4. I–V and failure characteristics of hybrid Ag/TBB crystals corresponding to the resistance values of 5.1 Ω (a) and 200 Ω (b). Note the „twitching effect“ (alternating reconnection and disconnection) of the crystal before it is detached from the circuit.

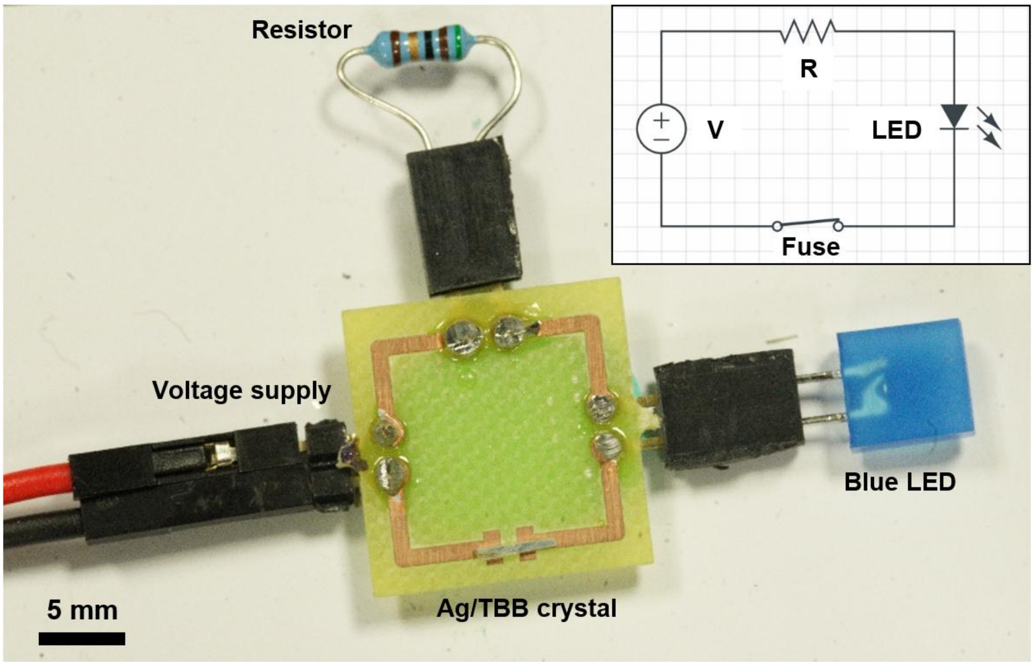

Figure S5. Electrical circuit used for testing of the Ag/TBB crystals as electrical fuse links. The inset shows a schematic of the circuit.

Legends to the supplementary movies

Supplementary Movie 1. Delayed thermosalient response from Ag/TBB crystal (dark-shaded crystal) relative to uncoated TBB crystal (light-shaded crystal).

Supplementary Movie 2. Delayed thermosalient response from Ag/TBB crystal (dark-shaded crystal) relative to uncoated TBB crystal (light-shaded crystal).

Supplementary Movie 3. Delayed thermosalient response from Ag/TBB crystal (dark-shaded crystal) relative to uncoated TBB crystal (light-shaded crystal).

Supplementary Movie 4. Thermographic recordings of the thermosalient transition of uncoated TBB crystal and coated Ag/TBB crystal.

Supplementary Movie 5. Thermographic recordings showing the heating and detachment of coated Ag/TBB crystals from the electrical circuit. The values on the right are the current and the voltage as the sample is heated.

Supplementary Movie 6. Thermographic recordings showing the heating and detachment of coated Ag/TBB crystals from the electrical circuit. The values on the right are the current and the voltage as the sample is heated.

Supplementary Movie 7. Thermographic recordings showing the heating and detachment of coated Ag/TBB crystals from the electrical circuit. The values on the right are the current and the voltage as the sample is heated.

Supplementary Movie 8. Demonstration of the use of coated Ag/TBB crystals as fuse links. The heated crystals undergo phase transition whereby they are detached from the corcuit and the current flow is interrupted, as observed with the LED indicator.

Supplementary Movie 9. Demonstration of the use of coated Ag/TBB crystals as fuse links. The heated crystals undergo phase transition whereby they are detached from the corcuit and the current flow is interrupted, as observed with the LED indicator.

Supplementary Movie 10. Demonstration of the use of coated Ag/TBB crystals as fuse links. The heated crystals undergo phase transition whereby they are detached from the corcuit and the current flow is interrupted, as observed with the LED indicator.