

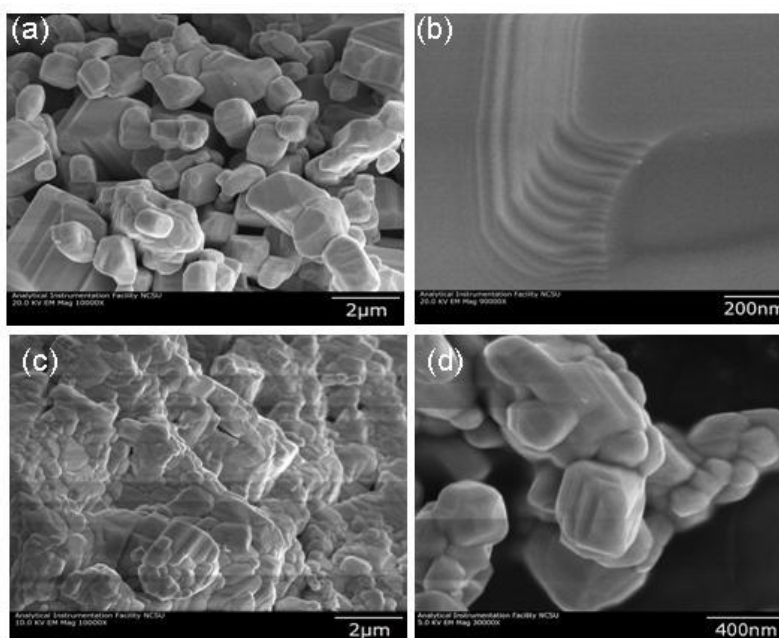
Title: New Molten-Salt Syntheses of Transition Metal Oxides with Photocatalytic Properties

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Metal-oxides particles with high photocatalytic activities can be synthesized via new molten-salt reactions. The $\text{La}_2\text{Ti}_2\text{O}_7$ and AgNbO_3 metal oxides were prepared in molten $\text{Na}_2\text{SO}_4/\text{K}_2\text{SO}_4$ and Na_2SO_4 fluxes at 1100 C and 900 C, respectively, for a wide range of flux:metal-oxide ratios (1:1, 2:1, 5:1, and 10:1 for $\text{La}_2\text{Ti}_2\text{O}_7$; 1:1, 2:1, and 3:1 for AgNbO_3) and reaction times (1 h, 2 h, 5 h, and 10 h for $\text{La}_2\text{Ti}_2\text{O}_7$; 1 h, 2 h, and 10 h for AgNbO_3). The related layered-perovskites RbLaM_2O_7 ($M = \text{Nb}, \text{Ta}$) were prepared in a molten RbCl flux at 1100 C for a wide range of flux:metal-oxide ratios (1:1, 2:1, 5:1, 10:1, and 20:1) and reaction times (2 h, 6 h, and 10 h). Starting from these RbLaM_2O_7 products, the ion-exchanged AgLaM_2O_7 compounds were prepared in a molten AgNO_3 flux heated to 250 C for 12 h. High purity of the resultant products for each was confirmed by powder X-ray diffraction, and their optical bandgap sizes were measured to be 3.8 eV for $\text{La}_2\text{Ti}_2\text{O}_7$, 2.8 eV for AgNbO_3 , 4.0 eV for $\text{RbLaTa}_2\text{O}_7$, 3.3 eV for $\text{RbLaNb}_2\text{O}_7$, and 3.3 eV and 3.0 eV for the Ag-exchanged compounds $\text{AgLaTa}_2\text{O}_7$ and $\text{AgLaNb}_2\text{O}_7$. $\text{La}_2\text{Ti}_2\text{O}_7$ exhibited rectangular-platelet particle morphologies with dimensions ranging from 500-6,000 nm, while for AgNbO_3 cube and bar-shaped particle morphologies were observed with a distribution of sizes from 500-5,000 nm. Individual particle sizes decreased with increasing amount of flux used in their syntheses, including within the RbLaM_2O_7 family. However, the AgLaM_2O_7 compounds did not exhibit any discernible particle size trends. Photocatalytic activities for the production of H_2 were tested under UV light for $\text{La}_2\text{Ti}_2\text{O}_7$ and under visible light for AgNbO_3 . For $\text{La}_2\text{Ti}_2\text{O}_7$, maximal rates were observed for the products from a 2:1 flux: $\text{La}_2\text{Ti}_2\text{O}_7$ ratio and a 1 h heating time, which was nearly two times greater than the rate for the $\text{La}_2\text{Ti}_2\text{O}_7$ prepared by solid state methods. For AgNbO_3 , a maximum initial visible-light photocatalytic rate was observed for the products from a 3:1 flux: AgNbO_3 ratio and a 1 h reaction time. By contrast, the solid-state preparation of AgNbO_3 did not exhibit any detectable photocatalytic activity.



SEM Images of the flux-prepared AgNbO_3 photocatalyst.