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# Hydrothermal synthesis of 1-D layered titanate ($A_2Ti_nO_{2n+1}$-like) nanomaterials from natural ilmenite sand

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**Abstract** – It was demonstrated that ilmenite mineral sand, without any previous chemical treatment, can be used as a new low cost feedstock for synthesis of layered titanate ($A_2Ti_nO_{2n+1}$-like) nanobelts (diameters < 50nm) through alkali hydrothermal process following a commonly used synthesis route for Fe-free precursors, i.e. synthetic anatase and rutile (temperatures between 140°C and 170°C and 10M NaOH). Using these synthesis parameters titanate nanobelts are predominant in the final product, although mixed with Fe-rich phases: hematite ($Fe_2O_3$) nanoparticles and leaf-like nanocrystals (Na,Fe-titanate phase).

Since Kasuga's innovation work on the alkali hydrothermal synthesis of titanate nanotubes from synthetic anatase and rutile ($TiO_2$) precursors [1] some effort has been invested on research of the influence of different $TiO_2$ and Ti-based precursors on morphology, chemical composition and crystal structure of the final 1-D nanoproducts (nanotubes, nanowires or nanobelts). Search for low cost feedstock usable in hydrothermal synthesis of nanomaterials is mandatory in order to maintain this synthesis route cost effective. The most low cost precursors are natural mineral sands; nevertheless there are just very few reports on the use of such precursors for synthesis of nanomaterials. Here we described, for the first time, hydrothermal synthesis of 1-D titanates ($A_2Ti_nO_{2n+1}$-like) in nanobelt form directly from natural ilmenite mineral sands.

Natural ilmenite sand unmilled and milled for 60min and 90min was hydrothermally treated in 10M NaOH solution at temperatures in the range of 120°C to 170°C. The products were analyzed by transmission electron microscopy and X-ray diffraction.

TEM of the samples synthesized at 140°C shows the predominance of leaf-like crystals (Fig. 1) having Ti, Fe, Na and O as the principal elements. Apart from the leaf-like crystals, spherical nanoparticles were also present. In the temperature range of 150°C to 170°C the formation of nanobelts with the diameters lower than 50nm is observed. However, the product is multiphasic, presenting three different particle morphologies (leaf-like nanocrystals and $Fe_2O_3$ nanoparticles besides the nanobelts) as can be seen in Fig. 2. Further increase of synthesis temperature leads to the formation of $Na_xFe_xTi_{2-x}O_4$ compound with $CaFe_2O_4$ crystal structure and arrow-like crystal morphology.

It was shown that ilmenite mineral sand, without any previous chemical treatment (such as for example acid leaching), can be used as a new low cost feedstock for synthesis of layered titanate ($A_2Ti_nO_{2n+1}$-like) nanobelts (diameters < 50nm) through alkali hydrothermal process following a commonly used synthesis route for Fe-free precursors. Using these synthesis parameters titanate nanobelts are predominant in the final product, although mixed with Fe-rich phases: hematite ($Fe_2O_3$) nanoparticles and leaf-like nanocrystals (Na,Fe-titanate phase).

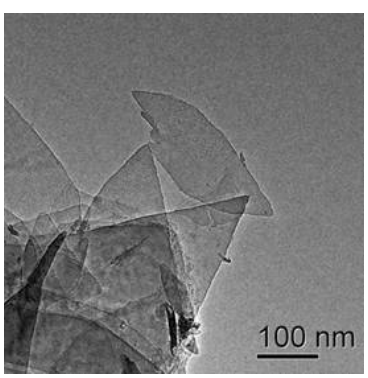


**Figure 1:** TEM image of leaf-like crystals obtained at 400°C.

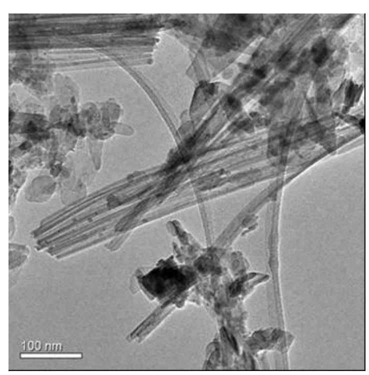


**Figure 2:** TEM image of the product obtained at 170°C.

[1] T. Kasuga, M. Hiramatsu, A. Hoson, T. Sekino, K. Nihara, Langmuir 14 (1998) 3160.