Microstructure of Mo-V-Te-Nb mixed metal oxides for selective oxidation of propane to acrylic acid

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Introduction

Selective oxidation of propane to acrylic acid has been studied widely over the last few decades. Among the catalysts proposed for this reaction, Mo-V-Te-Nb oxides have been found to be among the most promising. These materials have been reported to be able to give yields of acrylic acid above 40% [1]. The preparation method and thermal pretreatment conditions in the production of these catalysts have proven to be crucial for the activity of the final catalyst [2]. Although it has been proposed that the most active catalysts are produced by precipitation and spray drying, highly active catalyst can also be synthesized by hydrothermal synthesis [3].

Due to the complexity of these catalysts, preparation and characterization are formidable tasks. Small variations in preparation parameters and elemental stoichiometry, can result in grossly different products. Thus a high degree of control is needed in the preparation phase. To ensure homogeneity of the final product, several characterization techniques are employed throughout the preparation route.

Materials and Methods

In this work, Mo-V-Te-Nb oxides have been prepared by hydrothermal synthesis under varying conditions. The materials were characterized at each step of the preparation route using scanning electron microscopy (SEM), transmission electron microscopy (TEM), image simulations, and energy dispersive X-ray spectrometry (EDX). From these techniques, the morphology, phases and elemental composition were determined.

Results and Discussion

SEM of the phase pure catalyst revealed that the material mainly crystallized in a high aspect ratio needle-like morphology. Phase mixtures also showed these needle-type structures, but other morphologies were also observed. Thus the neddle-type morphology is attributed to the M1 phase of the Mo-V-Te-Nb oxide.

The elemental composition and homogeneity of the calcined catalysts were found to depend strongly on the homogeneity of the precursor. EDX spectra were acquired at different positions on the materials. Each spectrum was quantified and the mean value determined. The standard deviation of this value expresses the spatial homogeneity. Precursors showing a high degree of spatial inhomogeneity in the elemental distribution resulted in catalysts containing two or more phases whereas precursors showing a high degree of elemental homogeneity resulted in phase pure catalysts.

The phase pure materials were examined in the TEM to locally determine the phase of the material. Assisted by high resolution image simulations, it was determined that all the needle-type structures could be indexed as being of the M1 phase. TEM also revealed that the needles consisted of single crystals as the lattice fringes progressed throughout the structure. EDX in the TEM revealed that the Nb content based on the metals (20%) is significantly increased compared to the Nb content in the reported structural analysis of the M1 phase (9%) [4].

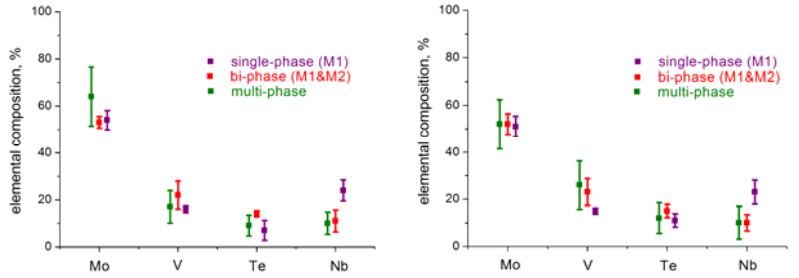

Figure 1: Quantified EDX data for precursors and the resulting catalysts.

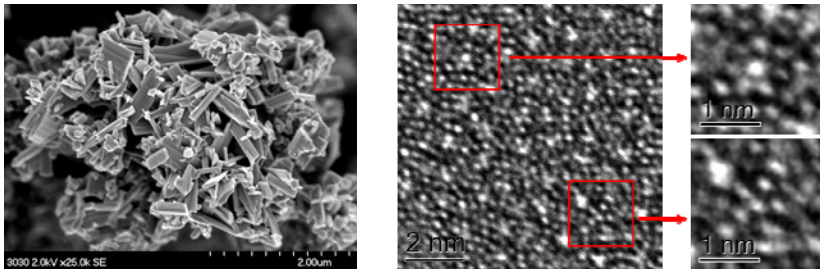

Figure 2: SEM and TEM of phase pure M1 material.

Significance

The results provide detailed information on the synthesis parameters of the Mo-V-Te-Nb oxides. Characterizing each step of the preparation route, determines the sensitive of a given step on the nature of the final product.

References

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