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Time-resolved in situ Studies on the Formation of Molybdenum Suboxides during Reduction of MoO_3

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Reduction of MoO_3 with hydrogen (5 - 100 vol-%) in the temperature range from 573 K to 823 K was studied by in situ X-ray diffraction and X-ray absorption spectroscopy (XAS). The experiments performed focused on elucidating phase composition and evolution with time under isothermal as well as temperature programmed reduction conditions. At reaction temperatures below 723 K the reduction of MoO_3 to MoO_2 is a one-step process. At reduction temperatures above 773 K and H_2 concentrations higher than 10 vol-%, Mo metal is the final product of the reduction of MoO_3. In addition, at temperatures higher than 723 K the formation of Mo_4O_{11} was observed. However, Mo_4O_{11} is not an intermediate of the reduction of MoO_3 but is being formed in a parallel reaction from MoO_3 and MoO_2 at temperatures above 723 K. Quantitative XRD analysis revealed a sigmoidal shape of the evolution of MoO_3 and MoO_2 phases during reduction of MoO_3 and an increase in the crystallite size of the phases present. This Oswald ripening indicates a nucleation-growth kinetic to govern the reduction of MoO_3 under the conditions studied. The results presented in this work clearly demonstrate the potentials of a combined application of in situ XRD and XAS to reveal phase composition and kinetics of solid state reactions.