Review of PhD thesis:

Mgr. Václav Valeš
High-temperature X-ray Diffractometry of Thin Layer

Submitted PhD thesis deals with the study of the crystallization and structural transformation during thermal treatment of the systems consisting of Fe, Ti, and Ce oxides. It is focused mainly on three main topics: Fe_2O_3 nanoparticles, Fe_2O_3-TiO_2, and CeO_2 – TiO_2 mixed oxides system. Last minor part deals with the self-ordering of nanoparticles for graphen modification. Respective physical properties were studied mainly using X-ray powder diffraction and other X-ray scattering methods.

The extent of this thesis corresponds to the studied systems. The work has 114 pages with relatively dense text. I must appreciate mainly publication activity, Václav Valeš has published 21 papers what is much more than is generally found in the publication activity of other PhD students. The thesis is divided into 6 chapters including Theory and Experimental and four chapters handling the results. The exhaustive list of 132 references is given at the end of this thesis.

The work is written in English with the relatively small amounts of grammatical mistakes.

I have following remarks and subjects to discussion concerning submitted thesis:

1) page 27 – The Curie temperature of gamma-Fe_2O_3 is commented to be 928 K. What is the source of this value? It is known, that pure gamma-Fe_2O_3 is transformed into alfa-Fe_2O_3 in the temperature range of 450-500 °C and for this reason, the Curie temperature cannot be determined.

2) page 33 – In the case of Fe_2O_3/TiO_2 nanocomposites is stated that maghemite nanocrystals were prepared by coprecipitation methods using Fe^{2+}/Fe^{3+} nitrates. This is not possible due to the fact, that anionts Fe(II) and nitrates cannot coexist in the solution because of their redox potential. Probably, different salts of iron were used.

3) page 33-34 and 53 – Forming gas has the strong reduction effect. The Fe_2O_3/TiO_2 layers were probably reduced at the given annealing condition and the metallic iron was formed. This can be seen in Figure 4.3.b in the case of the samples annealed at 800 and 1000 °C where the diffraction peak at 45 degree 2theta occurs. This peak probably corresponds to the most intense peak of alfa-Fe. What is about other characterization methods used in this chapter (EXAFS and Mossbauer spectroscopy) – did these methods also reveal the presence of metallic iron?

4) page 70 – Figure 5.6. – There is mismatch between Figure and the description of the Figure, anatas is probably shown in green colour and red colour corresponds to another phase (not anatas).

In spite of those remarks, I must state that this work has good level. This thesis fulfils the conditions set out for the defence of doctoral thesis and it can be taken for the PhD defence procedure. I recommend, on the basis of this work, to confer the degree PhD.

Praha, May 31th, 2015

Daniel Nižňanský