

Ahmet Yavuz Oral
Zehra Banu Bahsi
Mehmet Ozer *Editors*

International Congress on Energy Efficiency and Energy Related Materials (ENEFM2013)

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Preface

The first International Congress on Energy Efficiency and Energy Related Materials (ENEFM2013) provided all scientists the opportunity to meet, present their work, discuss, and mutually interact in order to enhance and promote their research work.

This volume, published by Springer, includes all the papers presented at this Congress, held in Antalya, Turkey, October 9–12, 2013.

On behalf of the organizing committee we would like to thank all the plenary and invited speakers for their valuable contributions.

We would also like to thank TURA Tourism for their support in the organization of the Congress, as well as our sponsor Doga Nanobiotech Inc. Finally, we would like to thank the publishers for the quality of this edition.

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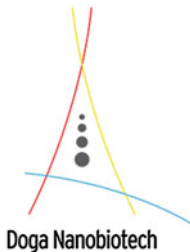
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Chapter 1

Study of the Effect of Transition Metals on Titanium Dioxide Phase Transformation

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Abstract MTiX samples with different atomic metal percentage were synthesised by sol-gel method and calcined at 400 °C under air. The anatase-rutile transformation in TiO₂ in the presence of transition metals (Cr, V and Mn) was investigated. The kinetics of anatase-rutile transformations were determined by XRD over the temperature range 500–800 °C. It was found that the presence of V and Mn accelerate the transformation anatase-to-rutile. However, the anatase phase stability increases as the chromium content increases.

1.1 Introduction

There are many studies on the anatase-to-rutile transformation [1–6]. This transition depends on several parameters such as the preparation techniques used, preparation conditions, thermal treatments, alkoxide nature, particle size, aging, nature and content of possible doping agents [7–11]. The doping metal oxides can be added by different ways such as further impregnations or mixed powders [12–14], but the best effect is obtained by a close contact between the stabilizing element and oxide. To optimize this interaction, many studies focused on doped TiO₂ prepared by various methods. Li et al. [15], have prepared titania-silica samples by sol-gel hydrothermal (SGH) method and sol-gel (SG) method. The results showed that the

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samples prepared by SGH had better thermal stability compared with the SG samples which retard the anatase-rutile transformation. On the other hand, Zhang [7] has used the titanium oxide doped with silica for photodegradation of toluene. It has found that the embedding of small amount of silica into anatase titania matrix enhanced the thermal stability of nanophase titania particle resulting in the suppression of the phase transformation from anatase to rutile phase. A clear understanding of the key factors that control the phase stability, the growth, and the phase transformation kinetics in materials is critical to quantification of materials behaviour. Consequently, new understanding of the factors that dictate the sequence of the phase transformations may provide insights into how phase composition, microstructures, and properties of titania-based materials can be manipulated [3]. In this, paper we report the sol-gel synthesis of MTiX mixed oxides with $X = 0, 5, 10$ and 20 of Cr, V and Mn atom percentage. We study the effect of the weight metals content on the temperature of the anatase-rutile transition and the kinetics of the transformation by in situ XRD.

1.2 Experimental

1.2.1 Synthesis Procedure

The preparation of MTiX was carried out in a 100 mL beaker. Titanium butoxide was dissolved in absolute ethanol. The mixture was stirred for 30 min with addition of an aqueous solution of metal and then a solution of ethanol and nitric acid was added drop ways in the mixture. A white gel was obtained which is dried and calcined in a muffle furnace at $400\text{ }^{\circ}\text{C}$ for 4 h under air atmosphere. The samples were called MTi5, MTi10 and MTi20 for 5, 10 and 20 of metal atom percentage respectively.

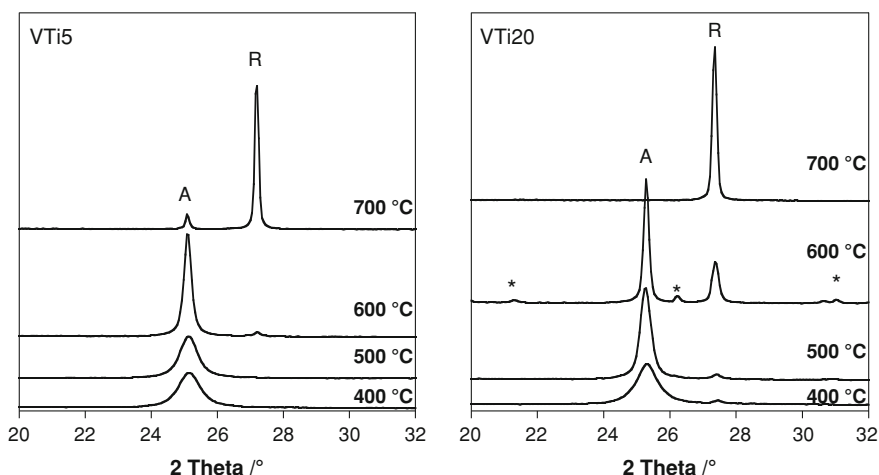
1.2.2 Samples Characterization

Micrometrics Tristar 3,000 instrument was using to measure adsorption-desorption isotherms at liquid nitrogen to determine specific area and calculation of mean pore size from BJH methods.

The crystalline phases of the samples were identified by X-ray powder diffraction (XRD) using Bruker D8-ADidentified VANTED diffractometers. The diffractograms were obtained under the following conditions: dwell time: 2 s; step: $0.04^{\circ} 2\theta$; divergence slit: 0.3° . The crystallite sizes were determined from the Scherrer equation [16] using the integrated width corrected from apparatus using LaB_6 as a standard.

Table 1.1 Surface area, porous volume and pore size of the samples

Sample	SBET m^2g^{-1}	Pore volume cm^3g^{-1}	Average pore size/nm
TiO ₂	68	0.06	3.60
CrTi5	180	0.22	5.00
CrTi10	230	0.19	3.30
CrTi20	152	0.10	2.60
VTi5	76	0.07	3.50
VTi10	129	0.11	3.50
VTi20	98	0.10	4.00
MnTi5	102	0.10	3.60
MnTi10	204	0.33	5.75
MnTi20	246	0.36	5.00

**Fig. 1.1** X-ray diffraction (XRD) patterns of VTiX; A anatase; R rutile; asterisk V₂O₅

1.3 Results and Discussion

1.3.1 Textural Data

The experimental results on BET surface area, mean pore diameter, and total pore volume of all synthesized materials are shown in Table 1.1. As obviously seen, the surface area increased with increasing of Mn content from 5 to 20 %. In the other hand the surface area increased with increasing both V and Cr content from 5 to 10 %. With increased Cr and V content (20 %), the observed loss in surface area could be attributed to probable segregation of chromium and vanadium [17–19]. The average pore size of anatase phase decrease with increasing of Cr content. In the case of VTiX samples, the average pore size remains constant with increasing of V content. Wearies an increasing of average pore size was observed with

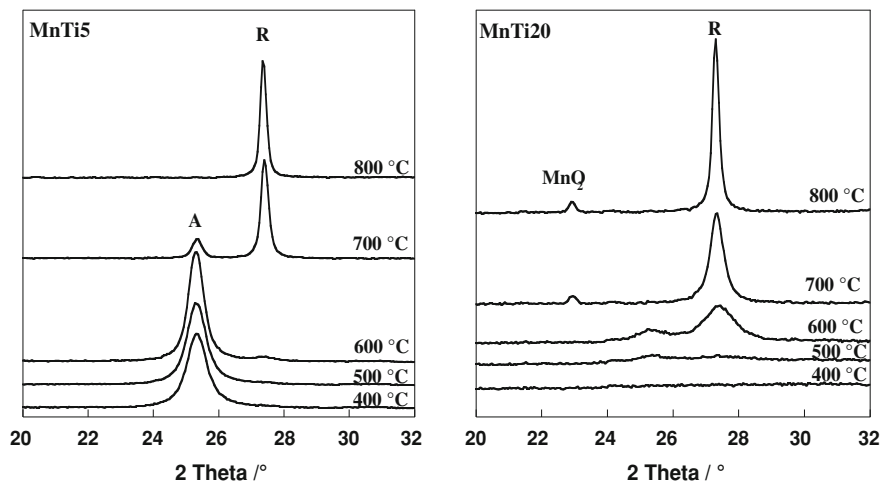


Fig. 1.2 X-ray diffraction (XRD) patterns of MnTiX; A anatase; R rutile

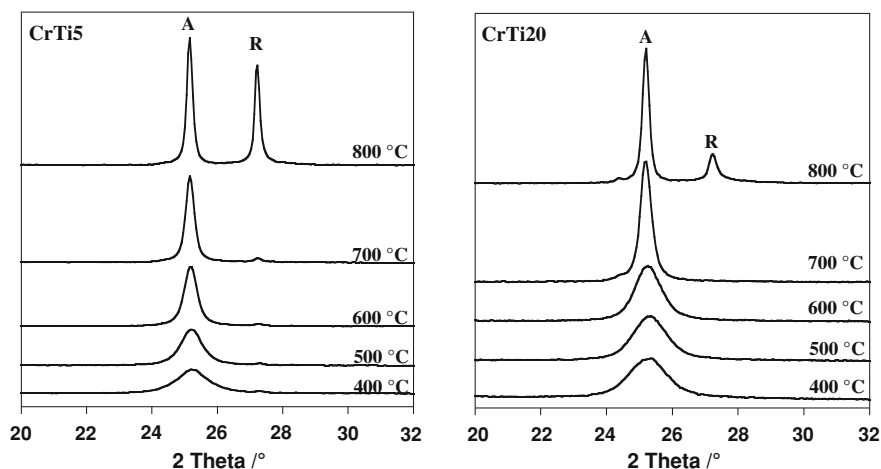


Fig. 1.3 X-ray diffraction (XRD) patterns of CrTiX; A anatase; R rutile

increasing of Mn content. The pore volume varied with varying metal and metal content. The biggest pore volume was signalled for MnTi20. So the presence of Mn content increased both pore size and pore volume.

1.3.2 X-ray Diffraction

The results of XRD analysis are shown in Figs. (1.1, 1.2 and 1.3). The diffractograms were recording in situ for MTiX samples. In this work we present only the

Table 1.2 Crystallite size versus temperature

Crystallite size (nm)											
T/°C		400		500		600		700		800	
Sample		A	R	A	R	A	R	A	R	A	R
TiO ₂		10	/	16	/	27	41	37	36	/	40
CrTi5		07	/	10	/	16	/	22	32	32	37
CrTi10		06	/	13	/	13	/	22	/	32	39
CrTi20		06	/	07	/	09	/	20	/	30	35
VTi5		09	/	11	/	24	34	41	42	/	47
VTi10		09	/	12	/	37	40	/	57	/	60
VTi20		10	/	19	28	36	31	/	40	/	41
MnTi5		12	/	12	/	15	/	24	31	/	37
MnTi10		11	/	11	/	14	13	/	25	/	36
MnTi20		/	/	/	/	18	09	/	18	/	35

diffractograms of MTi5 and MTi20. Anatase phase was observed for all samples at 400 and 500 °C excepted for MnTi20 with amorphous phase. The partial anatase-rutile transformation is at 600, 800, 600 and 600 °C for TiO₂ (not presented), CrTi20, VTi20 and MnTi20 respectively. The total transformation anatase-rutile is at 700, >800, 700 and 600 °C for TiO₂, CrTi20, VTi20 and MnTi20 respectively. However, the rutile fraction is higher for VTiX and MnTiX than TiO₂. The rutile phase is absent after treatment at 400–500 °C and decreases with increase of chromium content. Wearies the rutile fraction increases with increasing vanadium and manganese content. The Cr₂O₃ phase, MnO₂ phase and V₂O₅ phase appeared at 700, 600 and 700 °C for CrTi20, VTi20 and MnTi20 respectively. This last results leads that the segregation remains modest.

In Table 1.2 are illustrated the variation of the size of crystallite as a function of the temperature. For all samples a sintering of the anatase phase takes place with increasing of temperature from 400 to 800 °C. On the other hand, Cr content retards the sintering of the anatase phase whereas Mn and V content accelerate the sintering of the anatase phase. The anatase phase remains stable when the crystallite size is smaller than 14, 19 and 22 nm for MnTiX, VTiX and CrTiX respectively. This results are similar with results founded by Zhu et al [4], were the anatase phase is stable with small particle size wearies rutile phase is stable for particle size larger than 30 nm.

1.4 Conclusions

The MTiX mixed oxides with X = 0, 5, 10 and 20 of atomic metal content was synthesised by sol-gel method. All the samples were characterized by means of some techniques (XRD and BET). The metal content, temperature and particle size are crucial to the anatase phase stability and to the transformation anatase-to-rutile. Without addition of metal the anatase phase is stable at low temperature (400–500 °C). In the presence of chromium with different content, the anatase phase remains stable at 800 °C. Chromium ions incorporated in TiO₂ anatase phase delay the transformation to rutile and preserve smaller crystallite sizes thus inducing better catalytic activities such as hydrocarbon oxidation. The transformation anatase-to-rutile is accelerated with increasing both V and Mn content. The stability of anatase phase is favored with excess of Cr content and the crystallite size is less than 30 nm. Wearies the rutile phase is favored with V and Mn content.

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