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ISOTHERMAL ANALYSIS OF SiO_2 - TiO_2 GELS

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ABSTRACT: SiO_2 - TiO_2 samples have been prepared by the sol-gel method. We have studied the properties of gels, which were heat-treated at different temperatures. Fourier transform infrared (FT-IR) spectrometry was used to analyze the physical-chemical properties of gels. Thermal transformation of powders of these samples was studied by differential thermal analysis (DTA). We observed new binding by FT-IR in the range of 920 - 940 cm^{-1} and we assigned this band to Si–O–Ti bond.

KEY WORDS: thin films, sol-gel, thermal analysis, FT-IR

1. INTRODUCTION

Sol-gel process is now widely used to produce high performance, multi-functional, hybrid inorganic-organic materials providing structures, morphologies and properties that are inaccessible by conventional methods. Thin films prepared by sol-gel method either by dip coating or spin coating on various substrates are of increasing importance because of their optical, mechanical and electrical properties. Based films of titania-silica (SiO_2 - TiO_2) are of considerable interest due to their high thermal stability, high chemical durability, low thermal expansion coefficient, flexibly adjustable refractive index and high catalytic activity and selectivity. Condensation step is required in order to obtain a good quality film. This involves heat-treatment process.

Infrared (FT-IR) spectrometry has been employed to study of sol-gel materials, revealing many chemical and structural features of films.

DTA analysis was performed in order to determine the temperature of possible decomposition and phase changes of studied samples [1-3].

2. EXPERIMENT

SiO_2 - TiO_2 gels were prepared by mixture of SiO_2 and TiO_2 sols with various molar ratios (shown in Table 1). Individual sols were prepared by the scheme displayed on Fig. 1. Sols were dried at 80 °C to the constant weight. These xerogels were crushed on ultra-fine grained powders and then they were used on the FT-IR and DTA analysis.

Tab.1: The composition of gels (TEOS – tetraethylorthosilicate, $Ti(iPr)_4$ – titanium tetra-isopropoxide, IPA – isopropylalcohol, AcAc – acetylacetone)

(mol)	TEOS	$Ti(iPr)_4$	IPA	HNO_3	H_2O	AcAc
S	1	0	15.31	0.11	3.92	0
S2T1	2	1	51.45	0.99	8.44	2.12
S1T1	1	1	36.13	0.89	4.52	2.12
S1T2	1	2	56.95	1.67	5.12	4.24
T	0	1	20.82	0.78	0.60	2.12

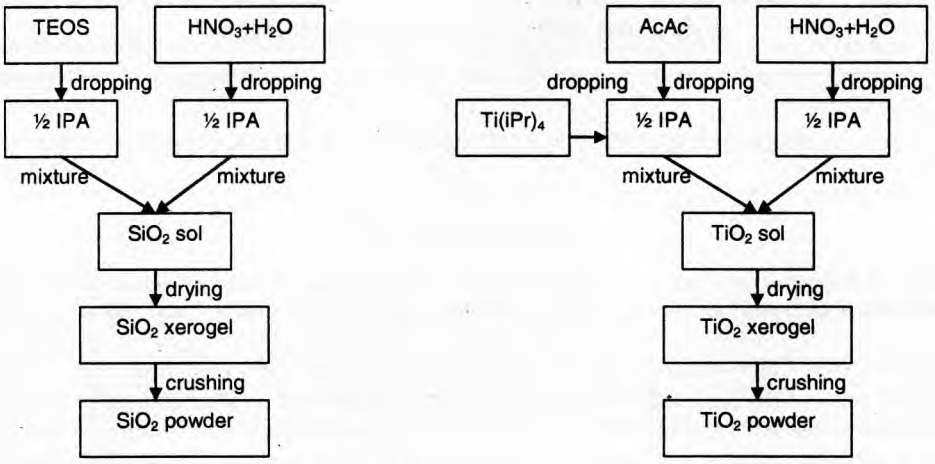

Fig. 1: The scheme of preparation of the SiO_2 and TiO_2 sol

Dried fine powders were subjected to differential thermal analysis (DTA, MOM Budapest, Hungary) to determine the temperature of possible decomposition and phase changes. Samples were heated at the rate of 10 °C/min at 400, 500 and 600 °C and holding time at each temperature was 120 minutes.

Samples' composition was analyzed by the Fourier transform infrared spectrometry (FT-IR, Bomem) in the KBr tablets form, in the range from 4000 to 400 cm^{-1} with 4 cm^{-1} resolution.

3. RESULTS AND DISCUSSION

Fig. 2, 3 and 4 show FT-IR absorption spectra of the individual samples at temperatures of 400, 500 and 600 °C.

The band at 800 cm^{-1} corresponds with vibration of Si–O–Si bond for the sample S. Low absorption band in the range of 700 - 800 cm^{-1} is attributed to vibration of Ti–O–Ti species for the T sample [3,4].

In the range of 920 - 940 cm^{-1} at the temperature 400 °C appears the band, which is assigned to created Si–O–Ti bond for the samples S1T1, S2T1, S1T2. This bond was probably established in the solution of sol. The Si–O–Ti bond doesn't move at the higher temperatures. This band can be overlapping the band which is assigned to Si–OH vibration from SiO_2. For the sample S, band at 950 cm^{-1} assigned to Si–OH vibration doesn't decrease as the temperature of heat-treatment of sample [4-6] increases.

Band at 1600 cm^{-1} appears in each sample and it is assigned to vibration of the molecular water. With the increasing temperature this band is decreased [5,6].

Fig. 5, 6 and 7 show isothermal DTA of the examined samples at individual temperatures. At the temperature of 400 °C for the samples with contents of TiO_2 (T, S1T2 and S1T1) there is no change in the behaviour of systems.

At the samples with higher contents of SiO_2 (S and S2T1), there is visible exothermic band, which increases its intensity with the increasing temperature. This is due to sintering of the particles in the systems.

At the temperature of 500 °C is for the samples with contents of TiO_2 clear, that the exothermic effect was formed. This effect occurs at the temperature of 600 °C too. This is probably due to crystallization of TiO_2 in the systems.

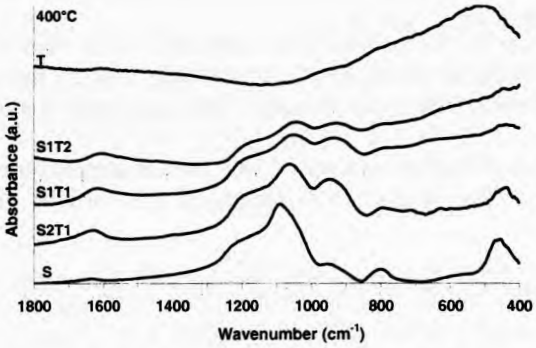

Fig. 2: FT-IR of samples at 400 °C

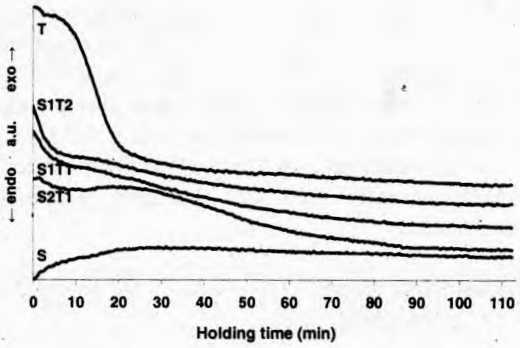

Fig. 5: DTA of samples at 400 °C

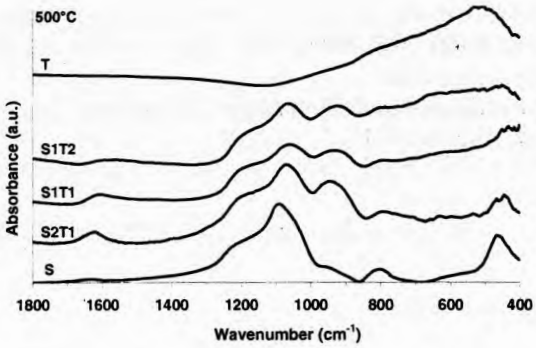

Fig. 3: FT-IR of samples at 500 °C

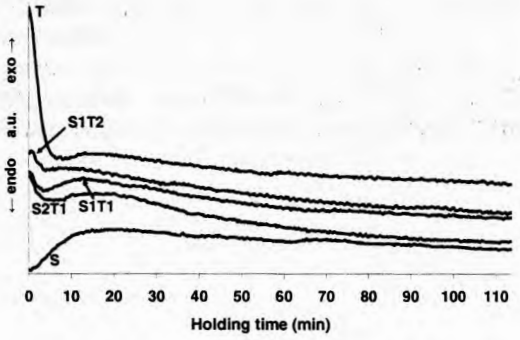

Fig. 6: DTA of samples at 500 °C

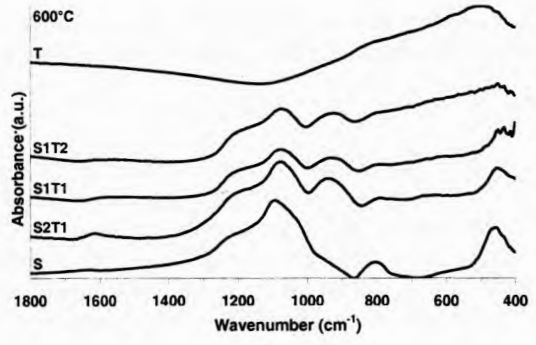

Fig. 4: FT-IR of samples at 600 °C

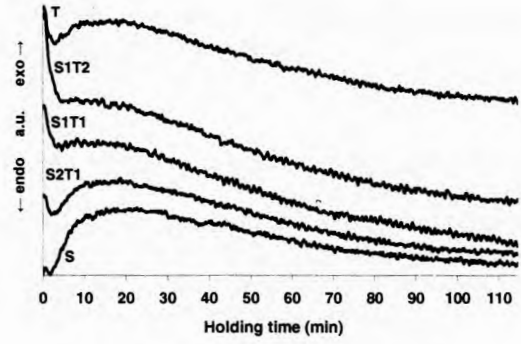

Fig. 7: DTA of samples at 600 °C

4. CONCLUSION

We prepared SiO_2 - TiO_2 sols by sol-gel method with various molar ratios. We have studied properties of gels heat-treatment at different temperatures. FT-IR was used to analyze the physical-chemical properties of prepared gels. We observed new Si–O–Ti bond in the range of 920 - 940 cm^{-1}

for the samples S1T1, S2T1 and S1T2. This bond was probably established in the solution of sol. The Si–O–Ti band doesn't move at the higher temperatures.

From isothermal DTA analysis we observed, that for the samples with contents of TiO_2 there is no change in the behaviour of systems at 400 °C. At the temperatures of 500 and 600 °C is for these samples with contents of TiO_2 clear, that the exothermic effect was formed. This is probably due to crystallization of TiO_2 in the systems.

The exothermic band is visible for the samples with higher contents of SiO_2 at each temperature, which increases with the increasing temperature. This is due to sintering of the particles in the systems.

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5. REFERENCES

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