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## **Coordination of the Titanium in Borosilicate Glasses by XRF, XPS and Raman Spectroscopy**

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COORDINATION OF THE TITANIUM IN BOROSILICATE GLASSES  
BY XRF, XPS AND RAMAN SPECTROSCOPY

**Keywords:** Coordination, Titanium, Borosilicate glasses,  
XRF, XPS, Raman.

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ABSTRACT

The coordination of Ti in borosilicate glasses have been studied by XRF, XPS and Raman spectroscopy. The results show that most of the Ti atoms are four-fold coordination in borosilicate glasses.

INTRODUCTION

The Ti atom is known to be four-, five-, and six-fold coordination by oxygen atoms in various crystal structures. A number of spectroscopic and diffraction investigations have been made elucidate the coordination and structural

role of Ti in Ti-containing glasses<sup>(1-4)</sup>. The present paper reports the results of the Ti coordination in borosilicate glasses.

## EXPERIMENTAL

### Compounds

Crystalline  $\text{Ba}_2\text{TiO}_4$  was prepared from certified grade anatase-type  $\text{TiO}_2$  and  $\text{BaCO}_3$  at 1300 C for 20 h. Its formation was confirmed by the x-ray diffraction technique. The Ti atoms in  $\text{Ba}_2\text{TiO}_4$  and anatase-type  $\text{TiO}_2$  are four-fold coordination and six-fold coordination, respectively.

Certified reagent grade  $\text{SiO}_2$ ,  $\text{BaCO}_3$ ,  $\text{H}_3\text{BO}_3$  and anatase-type  $\text{TiO}_2$  were used as the starting materials to obtain the glasses with the compositions given in Table 1. The starting materials were weighed, mixed in a roller-mill for several hours and put in a platinum crucible at 1300-1400°C for 1 h. The melt were quenched on steel plate. The plates of the glasses obtained were annealed at 550°C for some times and cooled to room temperature.

TABLE 1

Chemical Compositions of Borosilicate Glasses

Glass No.	Composition (wt%)			
	BaO	$\text{TiO}_2$	$\text{B}_2\text{O}_3$	$\text{SiO}_2$
G-1	30	10	20	40
G-2	30	10	30	30
G-3	30	20	10	40
G-4	30	20	40	10
G-5	30	30	10	30
G-6	30	30	30	10
G-7	30	40	10	20
G-8	30	40	30	0

TABLE 2  
Variance of TiK $\alpha$  from XRF Spectra

Compound	Variance (sq. deg.)	Compound	Variance (sq. deg.)
Ba <sub>2</sub> TiO <sub>4</sub>	0.12955	G-4	0.12986
TiO <sub>2</sub> *	0.13298	G-5	0.12967
G-1	0.12975	G-6	0.12941
G-2	0.12958	G-7	0.12951
G-3	0.12962	G-8	0.12989

\* anatase-type

### Instrumental

XRF spectra were obtained with a RIGAKU x-ray fluorescence spectrometer employing Cr target, TAP crystal and proportional counter. XPS spectra were obtained with a KRATOS x-ray photoelectron spectrometer employing Al x-radiation. Raman spectra were obtained with a SPEX RAMALOG laser Raman spectrometer employing Ar<sup>+</sup> laser device.

### RESULTS AND DISCUSSION

The results obtained from XRF spectra are given in Table 2.

It is seen from Table 2 that the variance of the TiK $\alpha$  of Ba<sub>2</sub>TiO<sub>4</sub> and anatase-type TiO<sub>2</sub> are 0.12955 sq. deg. and 0.13298 sq. deg., respectively. The variances of the TiK $\alpha$  of all Ti-containing glasses are in the vicinity of 0.12955 sq. deg.. It suggests that most of the Ti atoms in the borosilicate glasses are four-fold coordination.

TABLE 3  
Eb and Ek of  $Ti2p^{3/2}$  from XPS Spectra

Compound	Eb (eV)	Ek (eV)	Half-width of Ek (eV)
$Ba_2TiO_4$	457.9	1018.2	2.1
$TiO_2$	459.3	1020.9	1.75
G-2	458.3	1017.7	2.0
G-4	458.5	1018.3	2.1
G-6	458.4	1019.1	2.1
G-8	458.1	1020.0	2.15

\* anatase-type

The results obtained from XPS spectra are given in Table 3.

It is seen from Table 3 that  $E_b = 457.9$  eV,  $E_k = 1018.2$  eV and the half-width of Ek is 2.1 eV when Ti atom is four-fold coordination, whereas  $E_b = 459.3$  eV,  $E_k = 1020.9$  eV and the half-width of Ek is 1.75 eV when Ti atom is six-fold coordination. The  $E_b$ ,  $E_k$  and the half-width of the  $Ti2p^{3/2}$  of the borosilicate glasses are more close to 457.9 eV, 1018.2 eV and 2.1 eV, respectively. So most of the Ti atoms in the borosilicate glasses are four-fold coordination.

The results obtained from Raman spectra are given in Table 4.

It is seen from Table 4 that the Ti-O bonds of the borosilicate glasses are in the vicinity of  $754\text{ cm}^{-1}$ , the Ti-O bond of  $Ba_2TiO_4$ . It suggests that most of the Ti atoms in the borosilicate glasses are four-fold

TABLE 4  
Raman Spectra of the Crystalline and Glassy Compounds

Compound		Raman spectra (cm <sup>-1</sup> )		
Ba <sub>2</sub> TiO <sub>4</sub>		546m	754vs	1058w
TiO <sub>2</sub> *	394s	514m	638vs	
G-1			844s	1016w
G-2			850m	1108w
G-3			860m	1032w
G-4			856m	
G-5			840m	
G-6			838m	
G-7			824m	982w
G-8			832m	

\* anatase-type

coordination.

In a word, we may conclude that most of the Ti atoms in borosilicate glasses are four-fold coordination.

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