# Direct ultrasonic synthesis of classical high temperature ceramic phases at ambient conditions by innovative method

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Received March 05, 2013; Accepted May 18, 2013

It is well known that ultrasound waves accelerate chemical, physicochemical and electrochemical processes. In this respect, it deserves to investigate the applicability of this technique for synthesis of high tech materials with specific applications. Ferroelectric ceramics are used for production of various devices for electronic components like hightemperature capacitors, non-volatile memories (ROM), etc. The commonly used compounds for ferroelectric materials production are Bi<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, SiO<sub>2</sub> and Nd<sub>2</sub>O<sub>3</sub> and their different combinations. The classical route to obtain ferroelectric materials, particularly their high temperature specific phases, is to prepare a batch by weighing and mixing the precursors of the starting compounds in a hand mortar followed by high temperature melting usually over 1300°C which need higher class ovens and then tempering the molten compositions. This work presents a novel approach for synthesis of ferroelectric ceramic materials of the system Bi<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>-SiO<sub>2</sub>-Nd<sub>2</sub>O based on agitation of the precursors in powerful ultrasonic field generated by custom-made specialized device. The method is extremely simple and energy saving since it avoids using high temperature equipment. Applying this innovative approach the high-temperature phase Bi<sub>12</sub>TiO<sub>20</sub> having specific optical properties was synthesized at room temperature and verified by XRD analysis, optical microscopy and IR spectroscopy. The ultrasound agitation has pronounced effect on thermally treated samples too. Furthermope, studies are under way to establish the relationship between the mayor process parameters and the properties of the obtained ceramic materials (power of the applied ultrasonic field, temperature, duration, etc.). Our experiments show that this new useful method exhibits wide applicability in future hydrogen energy conversion systems, for optical, electro-optical and ferroelectric ceramic materials production. We suggest using it for production of other glass ceramics, superconductors, metal ceramics and composite materials, and also as an alternative or addition to the mechanochemical methods for both synthesis and activation.

Keywords: bismuth titanate ceramics, ultrasonic synthesis, electrical characteristics, microstructure, XRD

### INTRODUCTION

Aurivillius family oxides including Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> are of great interest in the last years due to their potential for electronic applications as transducers, capacitors, and acoustic piezo-sensors with high temperature piezoelectric properties, (high Curie temperature) [1, 2]. Many techniques have been employed for preparing a layered structure of bismuth titanate phases including powders and bulk ceramics: molten salt synthesis, co-precipitation, calcinations. sol-gel mechanochemical method and others. Between them the crystallization from melts or glasses [3-6] gives the possibility to control more easily the particle size distribution, morphology

In the previous studies [20, 21] it has been shown that the introduction of 20 - 40 mol% SiO<sub>2</sub> simulates the partial amorphization of the samples. The main established phases in super cooled melt

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crystallographic orientation. As it is well known the phase formation and the properties of these materials are strongly influenced by the method of preparation. On the other hand co-doped Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> ceramics are verv promising to commercialization. The introduction of Nd<sub>2</sub>O<sub>3</sub> as additive allows obtaining of materials with more effective electrical and dielectric properties, such as a higher remnant polarization and height resistance to fatigue [7-18]. The other challenge is the existence of solid solutions in the system Bi<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>-Nd<sub>2</sub>O<sub>3</sub>. By S. Kunej et al. [19] were described the solubility limits of three solid-solutions: Bi<sub>(1.6-</sub>  $_{1,08x}$ )Nd<sub>x</sub>Ti<sub>2</sub>O<sub>(6,4+0,3x)</sub>, (0,25<x<0,96), Nd<sub>2-x</sub>Bi<sub>x</sub>Ti<sub>2</sub>O<sub>7</sub>, (0 < x < 0.35), and  $Bi_{4-x}Nd_xTi_3O_{12}$ , (0 < x < 2.6).

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are either Bi<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> and Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> or only Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub>, in dependence of the cooling rate and composition. The other important result was that simultaneous introduction of SiO<sub>2</sub> and Nd<sub>2</sub>O<sub>3</sub> as additives [21] in bismuth-titanate ceramics changes the glassformation ability and electrical properties.

These results motivated us to continue our experiments in this field. The purpose of the present work is to prepare, by melt quenching method, polycrystalline or glass-crystalline materials in the system Bi<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>-SiO<sub>2</sub>-Nd<sub>2</sub>O<sub>3</sub> and to study their electrical properties depending on composition and temperature and to compare the results obtained after ultrasonication of precursors.

Beneficial effects at using ultrasound technology are described in [22, 23].

#### **EXPERIMENTAL**

The melting is performed in alumina crucibles at 500 °C to 1450°C according to way of preparation.

The samples are homogenized in two ways (Figure 1): 1) 15 minutes mixture; 2) 15 minutes mixture and then ultrasound homogenization (20 min).

The obtained samples with their batch compositions, visual observation and method of preparation are presented in Table 1 (obtained without ultrasound treatment) and Table 2 (obtained with ultrasound treatment).

Data for selected samples will be presented below. The samples, obtained by the first method, are heated in temperature range 1260 – 1450°C.

The samples, obtained by the second method, are heated in temperature range 500 – 1200°C.

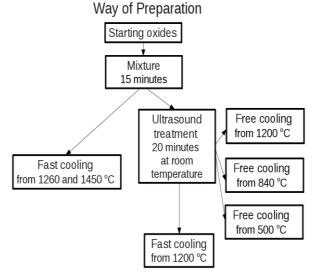


Fig. 1. Scheme of preparation of the samples.

#### RESULTS AND DISCUSSION

In this method ferroelectric materials are melted in alumina crucibles at  $1450^{\circ}C$  and super cooled to room temperature by pouring between two cooper plates. The detected main phases are  $Bi_4Ti_3O_{12}$ ,  $Bi_{12}TiO_{20}$  and  $\delta$ - $Bi_2O_3$ .

Samples were treated by ultrasound at room temperature which leads mainly to forming of phase  $Bi_{12}TiO_{20}$  (for samples J, L, 4D) and  $Nd_2O_3$  (for samples 6, 11). Subsequent thermal treatment shows the formation of the phase  $Bi_4Ti_3O_{12}$ , maintaining the phase  $Bi_{12}TiO_{20}$  (Figures 2-4).

**Table 1.** The obtained without ultrasound treatment samples with their batch compositions, visual observation and method of preparation.

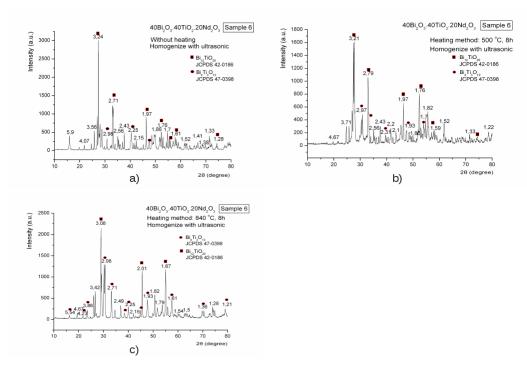
	Batch Composition	Phase formation according XRD	Method of Preparation
6	$40Bi_{2}O_{3}.40TiO_{2}.20Nd_{2}O_{3} \\$		Tm=1450°C
		$Bi_{4}Ti_{3}O_{12}, Bi_{12}TiO_{20}, \ \delta\text{-}Bi_{2}O_{3}.$	Fast cooling 1450°C, 15 min.
11	$50Bi_2O_3.40TiO_2.10Nd_2O_3$		Tm=1450°C
		$Bi_{4}Ti_{3}O_{12}, Bi_{12}TiO_{20}, \ \delta\text{-}Bi_{2}O_{3}.$	Fast cooling 1450°C, 10 min.
4D	$32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3$		Tm=1450°C
		Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub>	Fast cooling 1450°C, 10 min.
j	$40Bi_2O_3.40TiO_2.20SiO_2$		Tm=1260°C
		Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub>	Fast cooling 1260°C, 15 min.
1	$40Bi_2O_3.50TiO_2.10SiO_2$		Tm=1260°C
		$Bi_4Ti_3O_{12}$	Fast cooling 1260°C, 15 min.

**Table 2.** The obtained with ultrasound treatment samples with their batch compositions, visual observation and method of preparation.

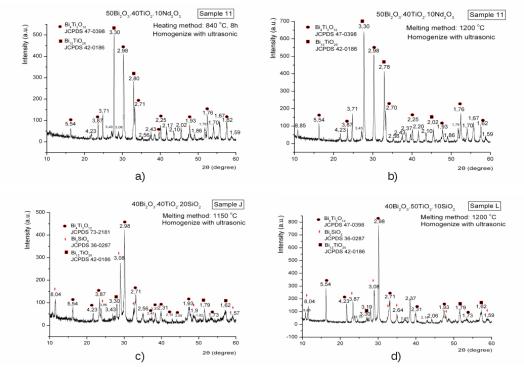
of prepar	ration.			
	Batch Composition	Phase formation according XRD		Method of Preparation
4D	$32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3$			Tm=840°C
		Bi <sub>12</sub> TiO <sub>20</sub> ,E	3i <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,Bi <sub>2</sub> SiO <sub>5</sub>	Ultrasound treatment Slow cooling 840°C, 8 hours
11	$50Bi_2O_3.40TiO_2.10Nd_2O_3$			Tm=840°C
		Bi <sub>12</sub> TiO <sub>20</sub> ,	Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,	Ultrasound treatment Slow cooling 840°C, 8 hours
11	$50Bi_2O_3.40TiO_2.10Nd_2O_3$			Tm=1200°C
		Bi <sub>12</sub> TiO <sub>20</sub> , Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,		Ultrasound treatment Slow cooling 1200°C, 15 min.
j	$40Bi_2O_3.40TiO_2.20SiO_2$			Tm=1150°C
		Bi <sub>12</sub> TiO <sub>20</sub> , Bi <sub>2</sub> SiO <sub>5</sub>	Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,	Ultrasound treatment Fast cooling 1150°C, 15 min.
j	40Bi2O3.40TiO2.20SiO2			Tm=1180°C
		Bi <sub>12</sub> TiO <sub>20</sub> , Bi <sub>2</sub> SiO <sub>5</sub>	Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,	Ultrasound treatment Slow cooling 1180°C, 15 min.
1	$40Bi_2O_3.50TiO_2.10SiO_2$			Tm=1180°C
		Bi <sub>12</sub> TiO <sub>20</sub> , Bi <sub>2</sub> SiO <sub>5</sub>	Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub> ,	Ultrasound treatment Slow cooling 1180°C, 15 min.
	32Bi <sub>2</sub> O <sub>3</sub> 40TiO <sub>2</sub> 20SiO <sub>2</sub> 8No Without Homoger Sign Sign Sign Sign Sign Sign Sign Sign	neating nize with ultrasonic 12-0186	3.30 1500 - 3.56 1000 - 3.56	32Bi_O_340TiO_220SiO_8Nd_O_3 Heating method: 500 °C, 3h Homogenize with ultrasonic  ■ Bi_TIO_3 JCPDS 42-0186 ■ Bi_TI,O_3 JCPDS 47-0398  1,76 1,97 1,67 1,52 1,33 1,28 40 50 60 70 80 29 (degree)
	Bi, Ti, O., JCPDS 47-0398 Bi, Ti, O., JCPDS 47-0398 Bi, Ti, O., JCPDS 42-0186 Bi, SiO, JCPDS 36-0287  400  400  400  400  400  400  400  4	840 °C, 8h		

 $\textbf{Fig. 2.} \ XRD \ patterns \ of sample \ with \ composition: a) \ 32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3 \ homogenized \ with \ ultrasound \ at \ room \ temperature; b) \ 32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3 \ homogenized \ with \ ultrasound \ and \ heated \ at \ 500°C;$ 

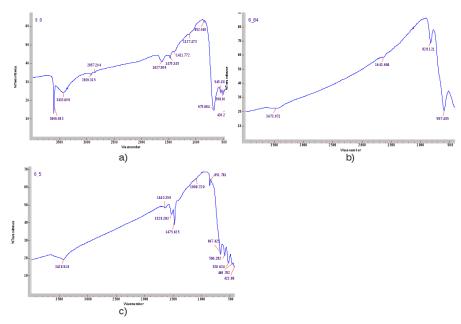
c) 32Bi<sub>2</sub>O<sub>3</sub>.40TiO<sub>2</sub>.20SiO<sub>2</sub>.8Nd<sub>2</sub>O<sub>3</sub> homogenized with ultrasound and heated at 840°C.



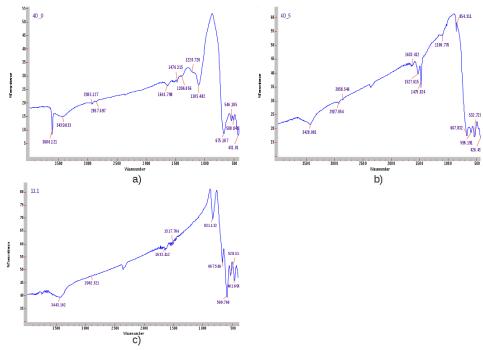
**Fig. 3.** XRD patterns of the external part of sample with composition: a)  $40Bi_2O_3.40TiO_2.20Nd_2O_3$  homogenized with ultrasound at room temperature;b)  $40Bi_2O_3.40TiO_2.20Nd_2O_3$  homogenized with ultrasound and heated at  $500^{\circ}C$ ; c)  $40Bi_2O_3.40TiO_2.20Nd_2O_3$  homogenized with ultra sound and heated at  $840^{\circ}C$ .



**Fig. 4.** XRD patterns of the external part of sample with composition: a)  $50Bi_2O_3.40TiO_2.10Nd_2O_3$  homogenized with ultra sound and heated at  $840^{\circ}C$ ;b)  $50Bi_2O_3.40TiO_2.10Nd_2O_3$  homogenized with ultra sound and heated at  $1200^{\circ}C$ ; c)  $40Bi_2O_3.40TiO_2.20SiO_2$  homogenized with ultra sound and heated at  $1150^{\circ}C$ ;d)  $40Bi_2O_3.50TiO_2.10SiO_2$  homogenized with ultra sound and heated at  $1200^{\circ}C$  and fast cooled.



**Fig. 5.** IR spectrum of sample with composition: a)  $40Bi_2O_3$ . $40TiO_2$ . $20Nd_2O_3$ ; b)  $40Bi_2O_3$ . $40TiO_2$ . $20Nd_2O_3$ ; c)  $40Bi_2O_3$ . $40TiO_2$ . $20Nd_2O_3$ .



**Fig. 6.** IR spectrum of sample with composition: a)  $32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3$ ; b)  $32Bi_2O_3.40TiO_2.20SiO_2.8Nd_2O_3$ ; c)  $50Bi_2O_3.40TiO_2.10Nd_2O_3$ .

The infrared spectroscopy of the investigated material gives some additional information concerning especially the connectivity of the short range building units as well some characteristic bands corresponding to the obtained crystal phases. Several bands are observed in the spectrum. Bands near 1000 cm<sup>-1</sup>, 800-900 cm<sup>-1</sup> as well as band near 470 cm<sup>-1</sup> are observed. This means that they may possess bridging bond Si-O-Si corresponding to the

band (above 1000 cm $^{-1}$ ) connected to partially polymerized SiO $_4$  tetrahedra. At 920 cm $^{-1}$  bridging bond Ti-O-Si decrease, the band below 920 cm $^{-1}$  due to Bi-O vibration of Bi $_2$ O $_3$ . At decrease of Bi $_2$ O $_3$  content this band shifts to higher frequency. At frequency 450 cm $^{-1}$  replacement of silica by Nd $_2$ O $_3$  up to 10% is quite interesting because the spectrum will be as similar as for pure Bi $_4$ Ti $_3$ O $_{12}$  for this compound. The typical band one is near 820

cm<sup>-1</sup>, the other band is near 580 cm<sup>-1</sup> and the following one is near 450 cm<sup>-1</sup>. Up to now there is no adequate explanation for the origin of this vibration (Figure 5, 6).

#### **CONCLUSIONS**

The investigation carried out confirms that depending on the condition of the melting and additional heat treatment of the super-cooled melt different poly-phase ceramic materials with various microstructures could be obtained in the system Bi<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>-SiO<sub>2</sub>-Nd<sub>2</sub>O<sub>3</sub>.

- 1. In the synthesized samples the presence of several phases including  $Bi_4Ti_3O_{12}$ ,  $Bi_2O_3$  polymorphs and  $Bi_{20}TiO_{20}$  are registered by melt quenching.
- 2. Samples treated by ultrasound without heating leads to formation mainly of the phase  $Bi_{12}TiO_{20}$  and of small amount of the phase  $Bi_4Si_3O_{12}$  (for samples with  $SiO_2$  presence in the batch composition) and  $Nd_2O_3$  (for samples with  $Nd_2O_3$  presence in the batch composition).
- 3. Subsequent heating of ultrasound treated samples (in temperature range  $500 1200^{\circ}$ C) shows the formation of the phase  $Bi_4Ti_3O_{12}$ , and of the phase  $Bi_{12}TiO_{20}$ .
- 4. The IR spectra confirms the degree of the connectivity between different types of the polyhedral building the short range order in the structure of the materials:
- a) The increased amount of  $TiO_2$  in glass ceramic material preserves the bridging bonds Si-O-Si and Si-O-Ti up to 40 %  $TiO_2$ .
- b) The increased amount of Bi<sub>2</sub>O<sub>3</sub> instead of SiO<sub>2</sub> leads to formation of isolated SiO<sub>4</sub> units.
- c) The introduction of  $Nd_2O_3$  does not change the IR spectra of the phase  $Bi_4Ti_3O_{12}$  up to  $10 \% Nd_2O_3$ .
- 5. Because of the proven efficiency of direct ultrasound synthesis (sonosynthesis) we can conclude it to be a very perspective technique for application in many cases like in glass, glass ceramics and ceramics pre-synthesis preparation where evaporation or sublimation of volatile components occur in uncontrolled manner and in this way the final chemical and respectively, the phase composition is changed. In particular cases the composition is very different from the initial one and researchers try to use various analytical methods to determine the composition. But all analytical methods have their natural limits, precision respective reproducibility. These efforts, as well as, expenses and time wasted could be avoided by applying the proposed ultrasonication method. The

analytical method appeared to be the analytical scales because 100% of the initial components are present in the final chemical and phase composition by using this method of preparation. One of the main problems in glass melting, as well as, in ceramics synthesis and sintering could be solved successfully by applying ultrasound treatment technique.

Acknowledgements: The presentation of the present research results has been supported by the Bulgarian Ministry of Youth, Education and Science, Project BG 051PO001-3.3.05/0001, cont. No. D002-170

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## ДИРЕКТЕН УЛТРАЗВУКОВ СИНТЕЗ НА ВИСОКОТЕМПЕРАТУРНИ КЕРАМИЧНИ ФАЗИ ПРИ ОБИКНОВЕНИ УСЛОВИЯ ПО ИНОВАТИВЕН МЕТОД

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Получена на 5 март, 2013; приета на 18 май, 2013

(Резюме)

Добре известно е, че ултразвуковите вълни укоряват химичните, физикохимичните и електрохимичните процеси. Във връзка с това си заслужава да се изследва приложимостта на тази техника за синтез на високотехнологични материали със специфични приложения. Фероелектричните керамики се използват за производство на различни електронни компоненти като високотемпературни кондензатори, ROM памет за компютри и други. Широко използваните съединения за производство на фероелектрични материали са Bi<sub>2</sub>O<sub>3</sub>, TiO2, SiO2 and Nd2O3, както и различни техни комбинации. Класическия начин на синтез за получаване на фероелекрични материали и особено високотемпературните им фази е да се приготви състав чрез претегляне и смесване на прекурсорите на изходните материали в ръчен хаван последвано от топене при висока температура обикновено до 1300°C. Това изисква по-висок клас пещи, след което следва темпериране на стопените композиции. Настоящата работа представлява нов подход за синтез на фероелектрични керамични материали от системата  $Bi_2O_3$ - $TiO_2$ - $SiO_2$ - $Nd_2O$  основан на облъчване на прекурсорите с високо мощно ултразвуково поле, генерирано от специализирано устройство изработено по поръчка. Методът е много лесен и е енергоспестяващ, тъй като се избягва използването на високотемпературно оборудване. С прилагането на този иновативен подход високотемпературната фаза Bi<sub>12</sub>TiO<sub>20</sub>, която има специфични оптически свойства беще синтезирана при стайна температура, което беше потвърдено с ренгенов фазв анализ XRD, оптична микроскопия и инфрачервена спектроскопия. Облъчването с ултразвук има изявен ефект и върху термично третираните образци. В ход са изследвания, които целят да се установи връзката между основните технологични параметри и свойствата на получените керамични материали (мощността на приложеното ултразвуково поле, температурата, продължителността и др.). Нашите експерименти показват, че този нов полезен метод има широка приложимост в бъдещите водородни преобразуватели на енергия, както и за производство на оптични, електрооптични и фероелекрични керамични материали. Ние предлагаме използването на този метод и за други стъклокерамични материали, свръхпороводници, металокерамични и композитни материали, а също така и като алтернатива или допълнение на механохимичните методи с цел синтез и активиране на материали.