Development ceramic composites based on Al_2O_3, SiO_2 and IG-017 additive

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Abstract. Based on high purity alumina and quartz powders and IG-017 bio-original additives the authors have developed new ceramic composite materials for different industrial purposes. The main goal was to fine a material and morphological structures of high performance ceramic composites as frames for development complex materials for extreme consumptions in the future. For this the mixed powders of **Al_2O_3**, **SiO_2** and **IG-017** bio-original additive were uniaxially pressed at different compaction pressures into disc shapes and were sintered in electric kiln under air (1) and nitrogrn (2) atmosphere. The grain size distributions of the raw materials were determined by laser granulometry. There thermo-physical properties were also determined by derivatography.

The prepared and sintered specimens were tested on geometrical sizes, microstructure and morphology by scanning electron microscopy, porosity and water absorption. In this work the authors present the results of their research and investigation.

1. Introduction

In our days the industry requires more and more low density and mechanically strong materials capable to work in high temperature environment. To satisfy these inquires more and more ceramics [1-7] and ceramic matrix composites [8, 9] or cermets [10, 11]. To increase the wear resistance and live times of machine parts or machine tools the ceramic coatings and ceramic based composites are used in very often [12-14] Generally in cases when the machine parts and machine tools have to work under high mechanical loads and working temperatures the ceramic materials and ceramic composites are probably the best [15-18].

Our aims with this research work to create ceramic matrix composites and develop artificial meta-kaolin and mullite particles on the basis of different mixes of **Al_2O_3**, **SiO_2** and **IG-017** bio-original additives.

2. Materials and experiments

In our experiments we used alumina powders of **Nabalox 315**, silica flour from **Fehérvárcsurgó** and **IG-017** refined bio-original additives developed and produced by Igrex Ltd. From these **3** components **18** different mixtures were prepared as it is shown in **Table 1** and milled in laboratory planetary ball mill Retch PM 400 trough 20 minutes of each at 200 rotation/minute. During the grinding we did not take into consideration the mechano-chemical phase transitions occurring under fine comminution. Later in a further work the determination of bond index of crushing can be also interesting using the easy method described by Maja S TRUMIC and her coauthors [19].

There were prepared by **6** different mixtures in the following **3** main combinations of components: **I./** - Al_2O_3+IG-017, **II./** - SiO_2+IG-017 and **III./** - Al_2O_3+SiO_2+IG-017. From these **18** mixtures the

specimens were uniaxially compacted at **150** MPa pressures into shape of cylindrical discs with diameters of **20** mm. From each mixture we compacted by **10** specimens. The filling weights of specimens were **10** grams in each case. In spite of the same compacting pressures and filling weights we have got specimens of different heights (Figure 1) depending on material compositions of the used mixtures.

Table 1. The volumetric relationship between the components

Number of mixtures		I./						II./						III./					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
The composition in **m%**	**Al_2O_3**	90	80	70	60	50	40	-	-	-	-	-	-	53	49	46	43	40	38
	SiO_2	-	-	-	-	-	-	90	80	70	60	50	40	31	29	27	25	24	22
	IG 017	10	20	30	40	50	60	10	20	30	40	50	60	16	22	27	32	36	39

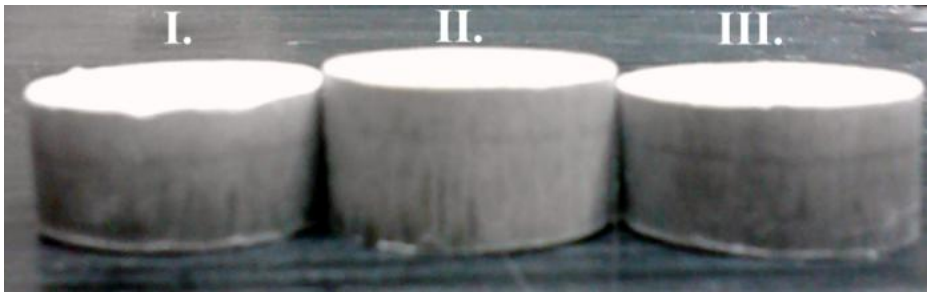


Figure 1. Typical differences in heights of compacted items

After compacting half of the specimens were fired (pre-sintered) in normal (**air**) atmosphere and the other half of the specimens were fired (pre-sintered) in nitrogen (N_2) by the firing curves as it is shown in Figure 2. In both cases the maximum sintering temperature was **1250°C**. This temperature is not enough high for formation of cristobalite crystals from tridymite phases in free **SiO_2** components of the mixtures.

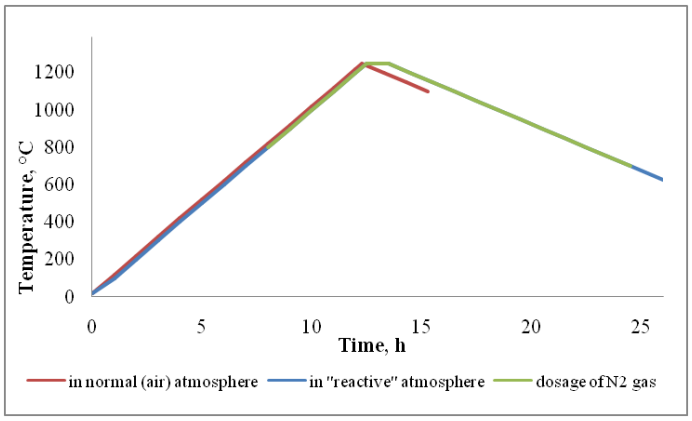


Figure 2. The sintering curves of specimens

3. Results and discussions

It is well known from practice that during sintering the **Al_2O_3 – SiO_2** ceramics items are shrinking and this shrinkage very strong depends on firing conditions and temperature [20-23]. Our experiments confirmed that the geometrical parameters of the pre-sintered ceramic items strong depend not only on the material compositions but on sintering conditions, including temperatures, pressures and atmospherically environments, thanking to the chemical reactions and phase transformations in solid stages and gasification or degasification of volatile component (Figure 3).

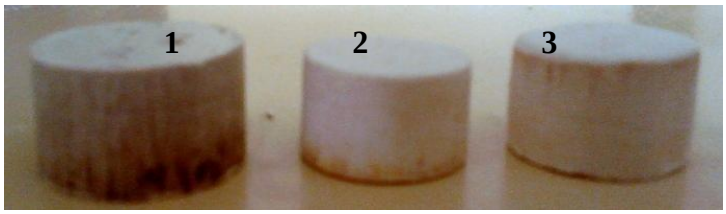


Figure 3. Changes of geometrical sizes of specimens depending on technological condition (1 - pressed green, 2 - fired in air, 3 - fired in N_2)

The results well represent that most of the bio-origin **IG-017** additives have fired out in normal (oxidation) atmosphere environment and the ceramic specimen suffered a large volume of shrinkage. This value of shrinkage is much higher than it could be expected because of the mullite formation from $\mathbf{Al_2O_3}$ and $\mathbf{SiO_2}$ components and phase transformation of the free $\mathbf{SiO_2}$ particles from β-quartz to α-tridymite crystals. At the same time in nitrogen ($\mathbf{N_2}$) atmosphere a swelling process can be observed thanking to the gasification and partly carbonization of the **IG-017** additive micro and nano particles. Depending on volume ration of the used bio-origin additive during pre-sintering (green firing) some of specimens have shrunk ("+") and some of specimens have swelled ("-") both in normal and nitrogen atmospheres as they are shown in Figure 4. In cases when the specimens were made from all three powders ($\mathbf{Al_2O_3}$, $\mathbf{SiO_2}$ and **IG-017**) we could observe only shrinkage in the normal atmosphere (1 green line) and only swelling (2 green line) in $\mathbf{N_2}$ atmosphere.

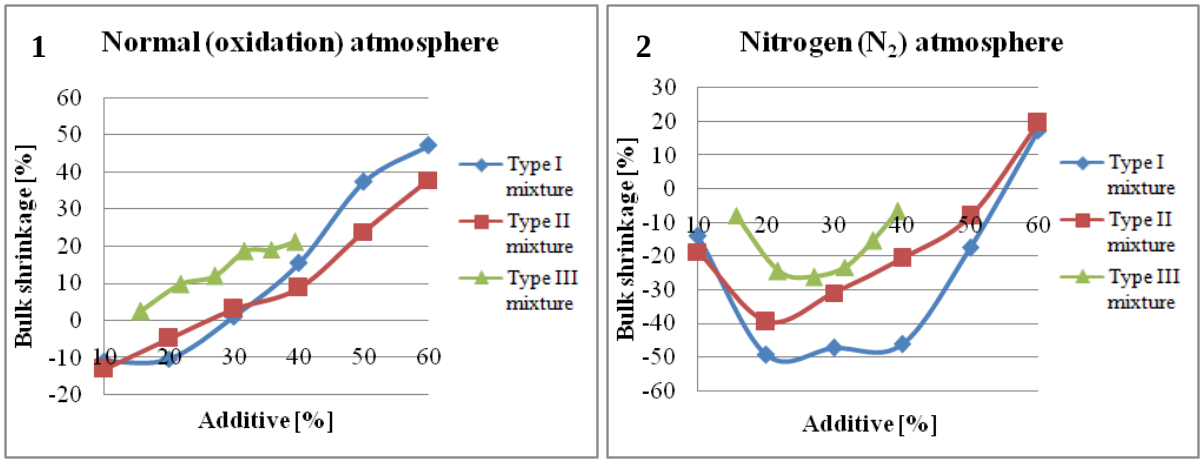


Figure 4. The change of volumes in normal (1) and in nitrogen (2) atmospheres

The burning weight losses (**I**) of specimens also were examined and determined by eq. 1.

$$I = \frac{m_1 - m_2}{m_1} \cdot 100 \quad [\%] \tag{1}$$

Where: the weight of the specimens in grams before firing ($\mathbf{m_1}$) and after firing ($\mathbf{m_2}$).
The burning weight losses of specimens are shown in Figure 5. The curves of this figure well illustrate that in cases when the portions of mixed **IG-017** additives were less than 30 m% the burning weight losses of specimens were much higher in nitrogen (2) than in normal (1) atmosphere.

The water absorption ($\mathbf{V_w}$) informs as about the open porous structure of the prepared ceramic items and can be determined as:

$$V_w = \frac{m_w - m_s}{m_w} \cdot 100\ [\%] \tag{2}$$

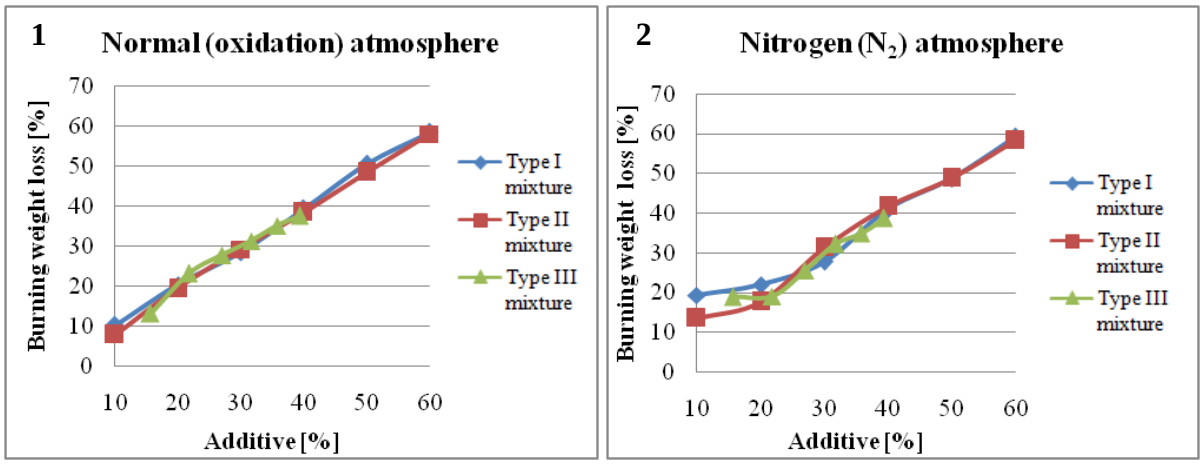


Figure 5. Burning weight losses during green firing of specimens
1 – in normal (oxidation) atmosphere, 2 – in nitrogen (N_2) atmosphere

Where: the weight of the specimens in grams before ($\mathbf{m_s}$) and after boiling ($\mathbf{m_w}$) in water through **2** h. The water absorptions of the green fired specimens as function of the volume of mixed IG-017 bio-origin additives are shown in Figure 6.

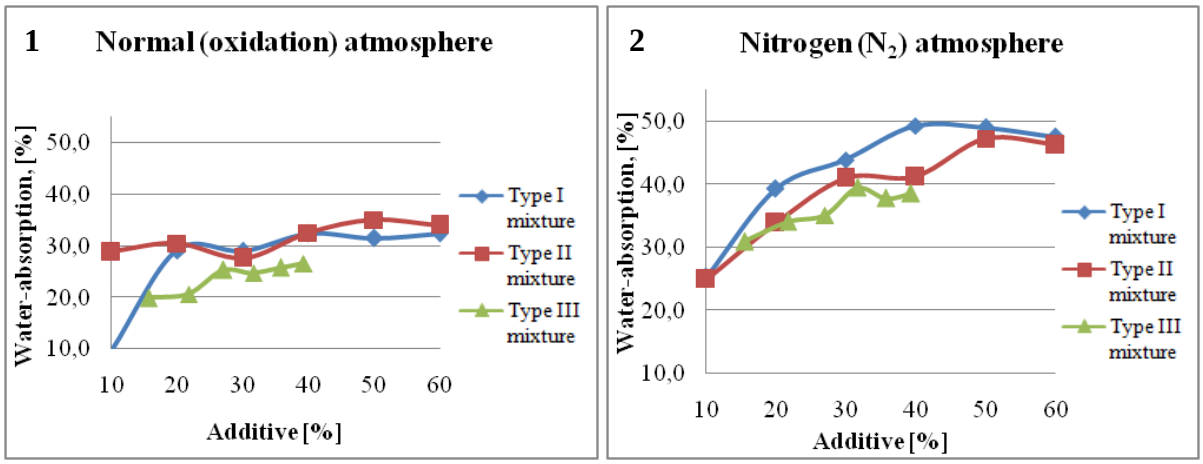


Figure 6. The water absorption of the green fired specimens
1 - fired in normal (air) atmosphere, 2 - fired in nitrogen (N_2) atmosphere

For the examination of the morphology and microstructure of the prepared specimens were used scanning electron microscopy and EDS. The typical microstructures and material compositions are shown in Figures 7-9 depending on quantity of the mixed **IG-017** additives and the firing conditions.

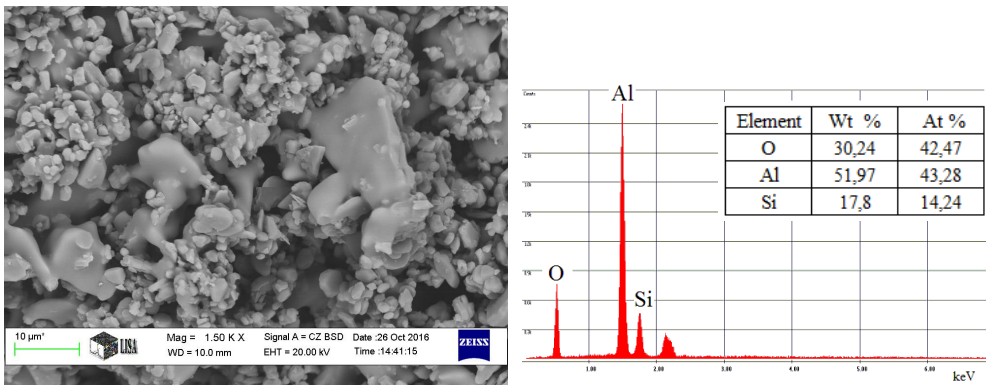
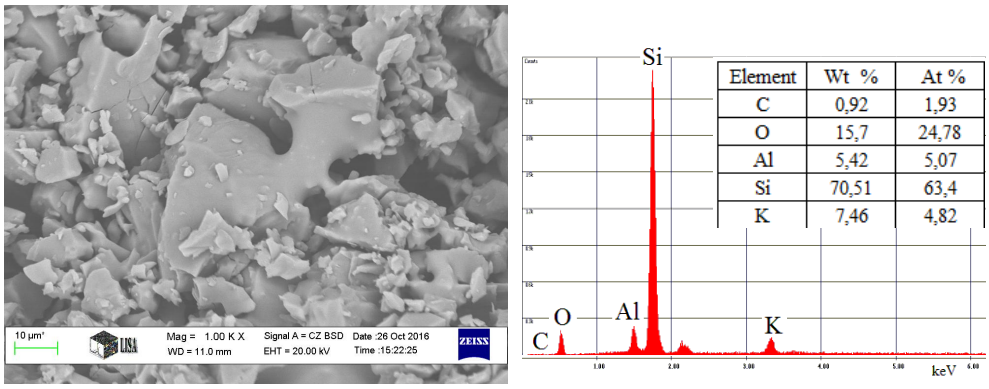
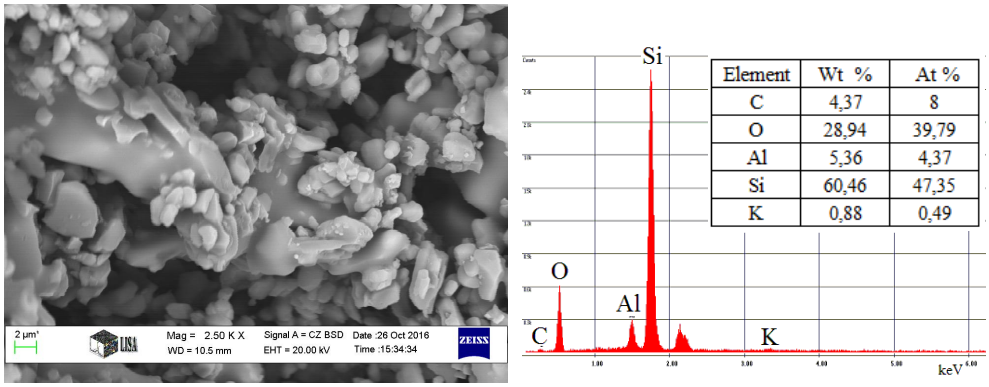


Figure 7. Specimen prepared from III/5 mixture and sintered in normal (air) atmosphere



Element	Wt %	At %
C	0,92	1,93
O	15,7	24,78
Al	5,42	5,07
Si	70,51	63,4
K	7,46	4,82

Figure 8. Specimen prepared from II/3 mixture and sintered in normal (air) atmosphere



Element	Wt %	At %
C	4,37	8
O	28,94	39,79
Al	5,36	4,37
Si	60,46	47,35
K	0,88	0,49

Figure 9. Specimen prepared from II/3 mixture and sintered in nitrogen (N_2) atmosphere

The SEM pictures of II/3 specimens well illustrate that the ceramic items pre-sintered in nitrogen (**N_2**) atmosphere have much more and larger pores than the items fired in normal (oxidation) atmosphere. At the same time the items pre-sintered in nitrogen have much more small crystalline particles thanking to the reactive sintering and carbonization of part of the **IG-017** additives and forming silicon carbide particles of micron and nano sizes. This formation of carbon base particles is well illustrated in EDS pictures, where the atomic percentage of carbon (**C**) has increased from **1.93** At% several times and achieved **8** At**%**. Thanking to the degradation of the **IG-017** bio-origin additives, the volume of oxygen also has increased forming a new material structure of **Si-O-C** in solid phase.

4. Conclusions

Using normal (air) and nitrogen (**N_2**) atmosphere for pre-sintering were developed high porosity new ceramic specimens from alumina (**Al_2O_3**) powder, quartz flour (**SiO_2**) and **IG-017** bio-original additives. The specimens were examined on shrinkage/swelling, water absorption and morphology. The achieved pore structures can make these ceramic materials suitable for high performance, light metal alloys impregnated new composites.

The formed new material structure of **Si-O-C** is much lighter than traditional **Al_2O_3-SiO_2** ceramics and at high temperature it has an increased contact angel of wetness for most of light metal melts. Probably the developed by us new ceramic composite material composition and is pore structure can be impregnated with light metals and metal-alloys more easy and more perfect than the traditional ceramics and ceramic composites.

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