Investigation the heat treatment influence on the morphology of electrospun mullite nanofibers

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Introduction

The use of microwave energy for processing materials has the potential to offer similar advantages in reduced processing times and energy saving. In conventional thermal processing, energy is transferred to the material through convection, conduction, and radiation of heat from the surfaces of the material. In contrast, microwave energy is delivered directly to materials through molecular interaction with the electromagnetic field. In heat transfer, energy is transferred due to thermal gradients, but microwave heating is the transfer of electromagnetic energy to thermal energy and is energy conversion, rather than heat transfer. As microwave can transfer energy throughout the volume of the material, the potential exists to reduce processing time and enhance overall quality.

Mullite fibers are outstanding refractory ceramic materials, which have excellent high temperature properties. Such as creep, heat and stress resistance, chemical stability and high temperature strength. Many successful methods have been reported for synthesis of ceramic fibers, ceramic nanofibers can be obtained via electrospinning.

Aim

After achieving very good results of synthesize pure and uniform mullite nanofibers, investigation of influence of heat treatment method on morphology and mullitization, was defined as the main aim. Therefore, electrospun mullite nanofibers were calcined at different temperatures in ordinary calcination and microwave furnace. Then via SEM images and XRD patterns, the role of heating method was investigated.

Materials & Methods

Starting materials used in the experiment were AIP ($C_9H_{21}O_3Al$), AN($Al(NO_3).9H_2O$) and TEOS($SiC_8H_{20}O_4$) which were purchased from MERCK Company and used as alumina and silica sources. PVA with average molecular weight of 7200(SigmaAldrich) was used as the polymeric template in the prespinning solutions. To obtain different mullite nanofibers, the resulting web of fibers was calcined at 800,1000,1200 and 1400 ℃ in calcination and microwave furnace.

Results & Discussion

The SEM images of mullite nanofibers before and after calcination at different temperatures, in the lower and higher magnifications, were shown in Fig 1. (in ordinary calcination furnace) and Fig 2. (microwave furnace). According to these images, it is observed that the calcined nanofibers have smooth surfaces after calcinations at 800,1000 and 1200℃(Fig. 1 & 2(a)–(d)), with a reduction in their diameter (around 100nm).The reduction of diameters is due to the burning-out of PVA, whereas the fibers after calcinations at 1400℃ showed rough surfaces with very small increase in their diameter which is due to the development of grain boundaries due to mullite crystallization. After calcination at 1200℃, due to further shrinkage of the fibers and complete burn-up of PVA, the diameter of the fibers decreased to the less than 100 nm. Fig.3 shows the comparison between calcined samples in calcination (a) and microwave (b) furnace. As shown in Fig.3 (b), because of microwave heating nature, samples have more thermal stability. Another influence of using microwave is on the mullitization starting temperature, as shown in Fig. 4. XRD patterns, prove that microwave method causes mullite nanofibers crystallized at lower temperature than those calcined in calcination furnace. It's due to nature of microwave, as explained in introduction part.

Conclusion

In this work, electrospinning was using for synthesis of fiber with small diameter and high homogeneity. In accordance with previous papers, precursor sol was prepared and electrospun. Then synthesized nanofibers sintered at different temperatures in calcination and microwave furnace. The details of crystal development, microstructure and thermal decomposition behavior of the electrospun nanofibers were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM). According to the results, sintered samples via microwave furnace had higher thermal stability and the crystallization temperature was lower than those sintered in calcination furnace.

Acknowledgements

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Graphs

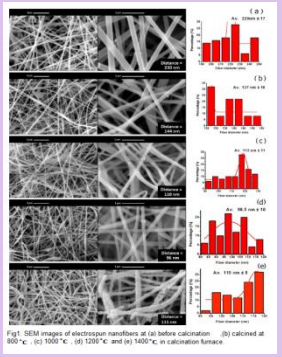

Fig1. SEM images of electrospun nanofibers at (a) before calcination ,(b) calcined at 800 ℃ , (c) 1000 ℃ , (d) 1200 ℃ and (e) 1400 ℃ in calcination furnace.

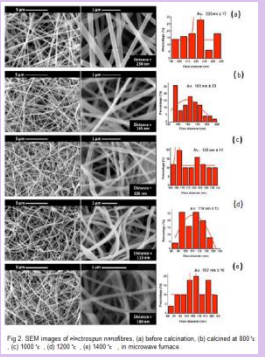

Fig 2. SEM images of electrospun nanofibers, (a) before calcination, (b) calcined at 800 ℃ , (c) 1000 ℃ , (d) 1200 ℃ , (e) 1400 ℃ , in microwave furnace.

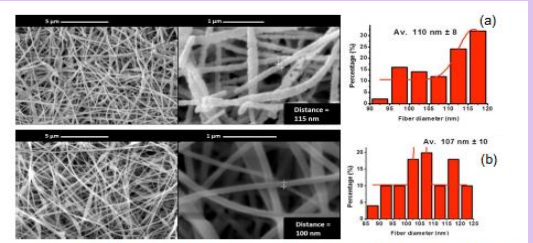

Fig 3. calcined samples at 1400 ℃ ,(a) in calcination and (b) microwave furnace.

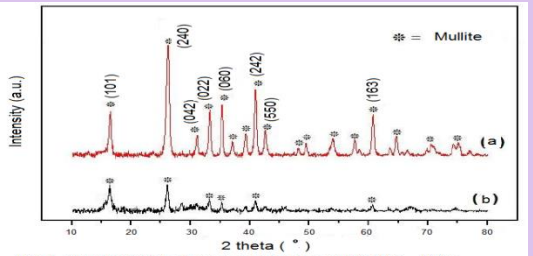

Fig 4. XRD patterns of calcined samples at 1000 ℃ , (a) in microwave and (b) calcination furnace.

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