Microwave-Assisted Synthesis and Characterization of Strontium Titanate Nanoparticles

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ABSTRACT

In microwave heating the total heating cycle time very less with comparison to conventional heating. This study is carried out to investigate the effect of the time duration of the calcination temperature on the structural properties of $SrTiO_3$ Ceramic powder. The present powder was synthesis by solid-state reaction method. The powders were calcined at a temperature of 1200^oC at four different durations, namely (20, 30, 40, and 50 minutes) using microwave sintering systems. The phase structure was investigated by using XRD along with Rietveld refinement analysis. A single-phase with a cubic structure was obtained in all calcined samples. The images and the particle size distribution of the calcined powder are carried out by using FE-SEM and particle size analyzer, respectively. The results show that the particle size has increased with an increase of the duration time, and the powder agglomerations' has taken place for the calcined at 50 minutes duration.

الملخص

في التسخين باستخدام المايكروويف يكون زمن الدوره الكلي قليل جدا مقارنة بالتسخين التقليدي. هذه الدراسة لتحديد تأثير الفترة الزمنية لحرارة التكليس على الخصائص التركيبية لمسحوق السيراميك $SrTiO_3$. تم تصنيع هذا المسحوق بطريقة اتفاعل الحالة الصلبة. المسحوق تكلس عند درجة حرارة 1200 درجة مئوية خلال اربع فترات مختلفة، بالتحديد (20، 30، 40 و50 دقيقة) باستخدام نظام المايكروويف المتمركز. تم تحديد فيز التركيب باستخدام XRD والانظمة التحليلة ذات العلاقة. احادي الفيز مع تركيب المكعب تم الحصول عليه في جميع العينات المتكلسة. تم الحصول على الصور وتوزيع حجم الجسيمات للمسحوق المتكلس باستخدام FE-SEM ومحلل حجم الجسيمات بالتوالي. النتائج أضهرت أن حجم الجسيمات أزداد مع زيادة الفترة الزمنية والمسحوق المتجمع ظهر بنسبة معنوية للتكلس عند فترة 50 دقيقة.

Keywords: *Strontium titanate, Calcination temperature, Crystallite size, Particle size. Particle* distribution

Introduction

Ceramics processing possesses a strong effect in the quality of the uniform body.It has an important role in obtaining its microstructure and final properties. It is known that the advance ceramics processing is generally carried out via high-temperature techniques such as conventional sintering and mould casting. The high-temperature processing of the

ceramics requires a high production cost and high energy utilization to the fabricate. Consequently, novel technologies defined as a nonconventional sintering method such as microwave processing are introduced to overcome the problem of cost and energy consumption while improving the properties of the obtained ceramics materials. In microwave heat treatment, the materials are coupled with microwave energy and absorb the electromagnetic energy and transform it into heat [1]. This process differs from the conventional technique. However, the heat is transferred to the sample by the conduction mechanism, radiation and conversion [1]. In the conventional calcination, the powder the heat starts from the surface and moving inward, which means that the gradient of the temperature takes place from the surface to enter the sample. However, in the microwave, the heat microwave radiation is incident into the materials, and the susceptor absorbs the radiation, and the heat uptake place [2]. Several advantages have been obtained from the microwave processing compared to conventional processing techniques such as increasing diffusion process, decreasing the consumption of energy, decreasing the processing time and decreasing the sintering temperature and improve the physical properties [3-7].

Several methods have been utilised to prepare SrTiO3 ceramics materials such as solid-state reaction [8], sol-gel [9], and coprecipitation [10]. All these techniques have some concern, such as longer time and high energy consumption and phase purity. In the present study, we have used microwave irradiation to assist heating of the starting materials with short time and low energy consumption, which can provide industry with the sustainable method in the future. The effect of microwave calcination on the crystal structures, microstructures and method were investigated. The obtained results conform that the microwave synthesis is a succes method for obtaining the desired microstructure with pure phase.

Experimental Section:

The solid-state reaction route with microwave heating of the starting materials has been used to synthesis the $SrTiO_3$ samples. The starting materials $SrCO_3$ (Sigma–Aldrich 99.99% purity) and TiO_2 (Sigma–Aldrich 99.98% purity) have been used to obtain the $SrTiO_3$ powder. The mixture was ball–milled for 10 hours duration. The mixture was kept in an oven furnace at 150^{o}C for drying. The drying powder was calcined using MWSS (microwave sintering system of ENERZI Microwave Systems, India with 1.4kW magnetron). The target temperature is kept at 1100^{o}C with a dwell time of 20, 30, 40 and 50 minutes and a heating and cooling rate of 50^{o}C/min. The calcined powders were once again re-milled for 10 h duration. The phase confirmation was done using XRD using a Bruker D8 X-ray diffractometer with Cu Kα wavelength (1.5418 Å). Fullprof refinement software was used to estimate the lattice parameters, atomic positions and unit cell volume. The morphological structure was characterized by using Field emission scanning electron microscopy (FE-SEM- Carl Zeiss, Ultra 55). Particle size analyzer was used to investigate the particle size.

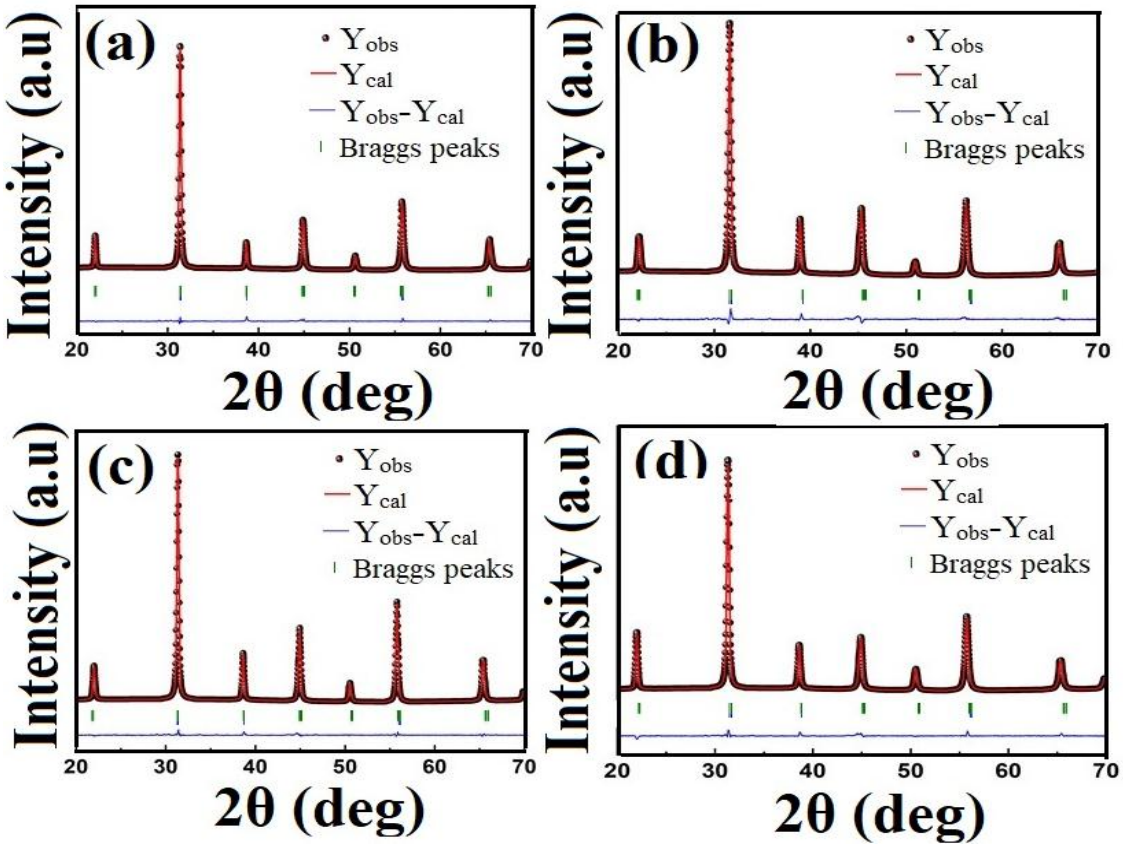

Fig.1: X-ray powder diffraction pattern of the $SrTiO_3$ obtained from Rietveld refinement analysis.

Results and Discussion:

The XRD patterns of the calcined $SrTiO_3$ powder is shown in Fig. 1. The Bragg reflections in the obtained patterns were indexed on a cubic unit cell with Pm3m space group [11]. The XRD patterns were also refined using Rietveld refinement software, as shown in Fig.1. The refinements were done to investigate the fine variation in the structural properties of different calcination temperatures. Each pattern in Fig.1 shows the XRD pattern which is obtained from the experiment, and the black line represents the theoretical fitting pattern and the blue line indicates data of difference error between the experimental pattern and the simulated one. The lattice parameters obtained after refinement of the XRD pattern for all samples are presented in Table 1. The established date shows a slight variation in lattice parameters and crystallite size with the increasing calcination time. The crystal structure of $SrTiO_3$ obtained from fullprof refinement is presented in Fig.2.

Table 1**:** The Rietveld Refinement Parameters of XRD data for the $SrTiO_3$ powders.

Calcination time (minutes)	20	30	40	50
Crystal system	Cubic	Cubic	Cubic	Cubic
Space group	Pm3m (No. 221)	Pm3m (No. 221)	Pm3m (No. 221)	Pm3m (No. 221)
Angles α=β=ɤ	90	90	90	90
Lattice constant a=b=c (Å)	3.9054	3.9065	3.9075	3.9087
Unit cell volume $(Å)^3$	59.56574	59.61609	59.66188	59.71687
Ch^2	2.12	2.33	2.24	2.01

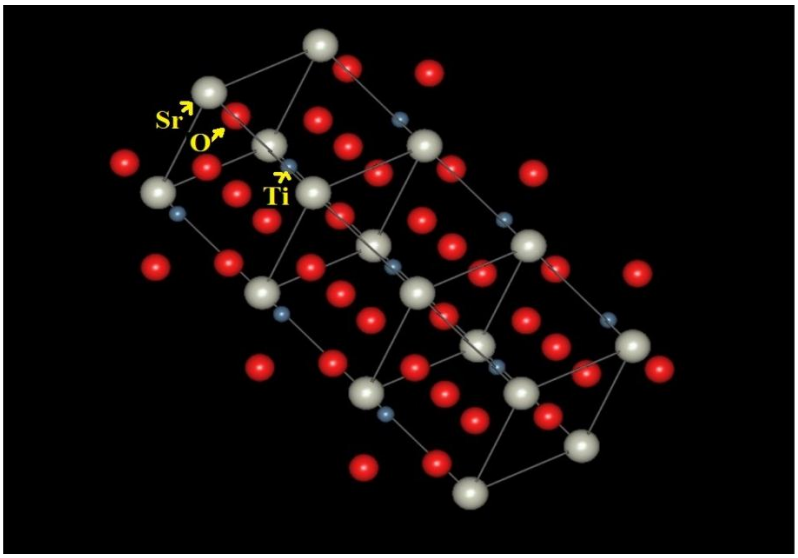

Fig. 2: crystal structure of $SrTiO_3$ obtained from fullprof refinement

The SEM images of the calcined $SrTiO_3$ powder at different durations are shown in Fig. 3. However, the particle size distributions of the corresponding images are shown in Fig.4. The images exhibit that the particle size was increased with increasing the dwell time and agglomerated at the dwell time of 50 min.

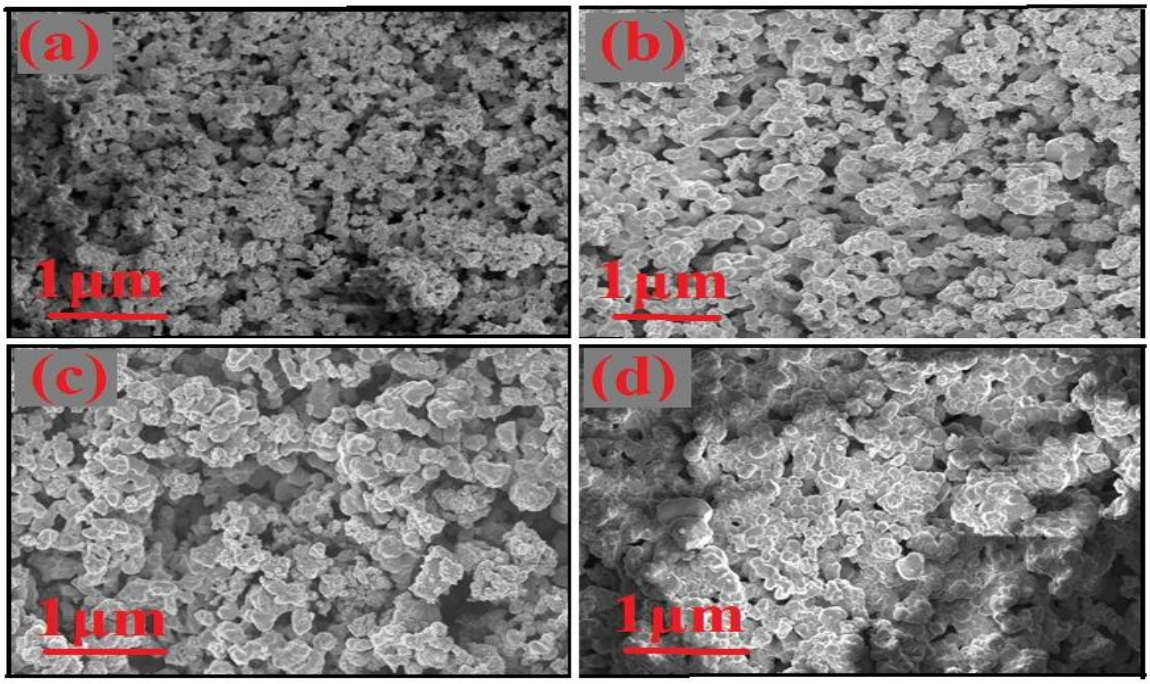

Fig. 3: The SEM images of the $SrTiO_3$ powder calcined by microwave system at durations of (a) 20 minute, (b) 30 minute, (c) 40 minute, and (d) 50-minute durations

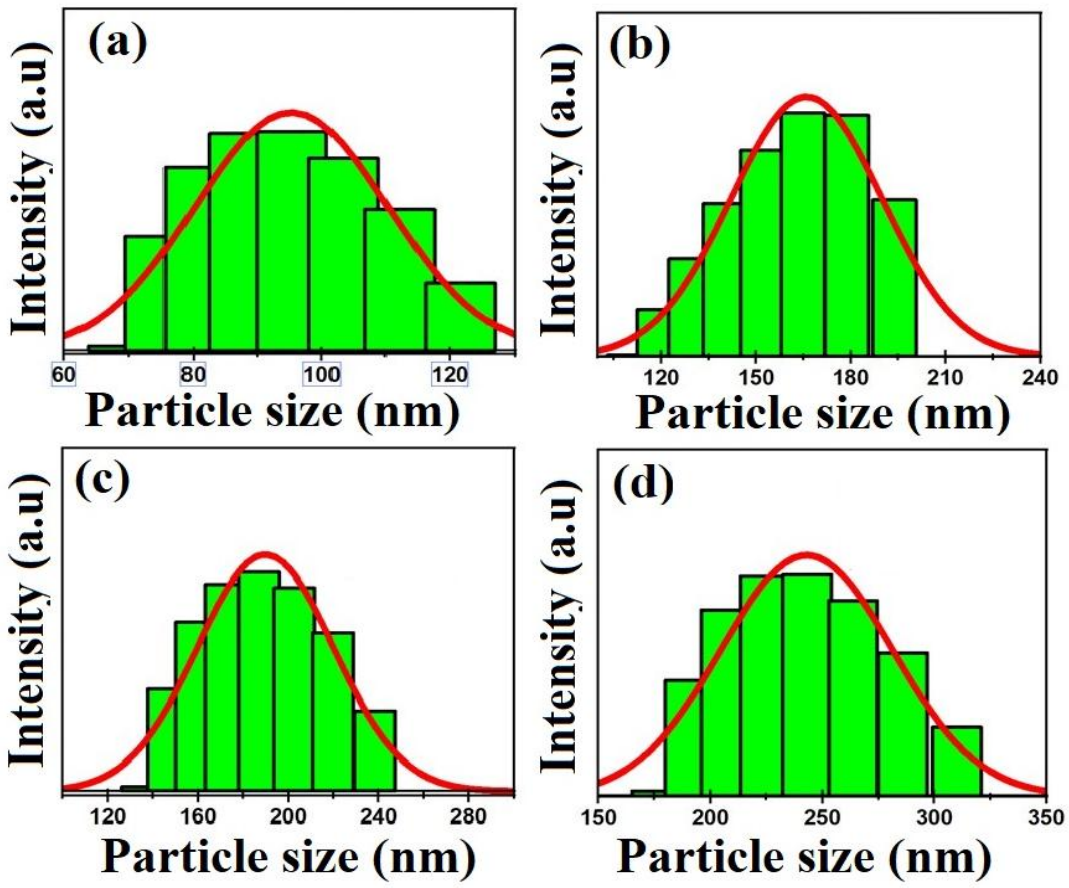

Fig. 4: The particle size distributions of the $SrTiO_3$ powder calcined by microwave system at durations of (a) 20 minute, (b) 30 minute, (c) 40 minute, and (d) 50-minute durations

. To investigate particle sizes, the obtained data from Particle size analysis has been fitted using Gaussian fitting. The particle sizes are observed to be a 95, 165, 189, and 243 nm when the calcination time is 20, 30, 40 and 50 minutes respectively. The particle size is larger, with an increase in the calcination dwell time. The phenomenon of growth the crystalline with increasing dwell time could be due to the enhancement of the crystalline volume to the surface ratio, which occurs due to particle size expansion [12-13]. For this reason, the microwave calcination of 1100 ◦C is considered as the optimum condition for preparation SrTiO3 nanoparticles with minimum energy consumption.

Conclusion

The effect of the time duration of the calcination temperature on the structural properties of $SrTiO_3$ nanopowder has been investigated. The nanopowders synthesis by solid-state reaction method and the calcination process has been carried out using microwave sintering systems. Single-phase structure was confirmed by XRD along with Rietveld refinement fitting. A single-phase with a cubic structure was obtained in all calcined samples. The images and the particle size distribution of the calcined powder are carried out by using FE-SEM and particle size analyzer respectivel, and the obtained results show that the particle size is increased with the increase of duration time and the powder agglomerations' is seen for the sample

calcined at the calcined at 50 minutes duration.

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