



## Liquid Phase Sintering of Nano Silicon Carbide Prepared from Egyptian Rice Husk Ash waste

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### Abstract

The aim of the present research is to prepare well dense sintered nano-SiC. Two samples of Nano-silicon carbide prepared by pyrolysis of rice-husk ashes as starting materials. The first was fine (hand-ground) rice-husk ash, and the second was prepared by ball milled technology. The firing temperature to obtain well crystalline nano SiC was 1550°C. The effect of pyrolysis under vacuum atmosphere had been tested. Herein the liquid phase sintering is used as a good sintering technique. The oxides sintering additives (3 wt% Y<sub>2</sub>O<sub>3</sub> and 7wt% Al<sub>2</sub>O<sub>3</sub>) play very crucial role in decreasing the apparent porosity and increasing the relative density of the sintered body if compared with that prepared without any oxide additives. Different sintering temperature firing range was applied here, and the optimum firing temperature that used to obtain well dense sintered SiC was 1950°C. With decreasing additive content, the consolidation of the materials became increasingly dependent on solid-state sintering. The microstructure underwent a transition from a microcrystalline matrix with nano-crystalline second phase (micro-nano) to a nano-crystalline matrix with nano-crystalline second phase (nano-nano) when the yttria and alumina content was lowered to 3 wt%.

Key words: Rice husk, Nano SiC, Metal oxides additives, Liquid phase sintering, Sintered nano-SiC.

### 1. Introduction

In Egypt, a large quantity of rice husk is generated as a by-product after rice harvest. Its major constituents are organic materials and silica. Silica is naturally distributed in the backbone cellulose structure of the rice husk [1], such that its content is more in the exterior compared with the inner walls. Not only it's burning creates pollution problems but the extremely fine silica ash is also toxic and thus constitutes a health hazard. Even careful incineration procedures cannot completely eliminate this air borne silica.

Silicon carbide seems to have the potential to satisfy the requirements in most of the applications as 'high temperature structural material' for the following important properties: superior oxidation resistance, superior creep resistance, right hardness, good mechanical strength, very high young's modulus, good corrosion and erosion resistance, and relatively low weight. It is important to mention here that the raw materials of silicon carbide are relatively

inexpensive (raw husk ash). Therefore, the final products are cost-competitive besides offering the advantages of superior technical performance over the other materials [2-4]. The researches on nanostructure SiC (nano-particles) are under extensive development. Nano SiC has many important applications are much greater than that of bulk SiC; which should be seriously considered for producing very high-strength ceramic composites. The combination of special mechanical, physical and chemical properties of SiC and nanostructure make nano size SiC an excellent candidate for designing the nano size devices.

The initial density of the green body is very important to achieve a higher sintered density [5]. After this stage, they have to be fired or sintered at sufficiently high temperature to complete different desirable phase transformations and also develop necessary atomic bonding or cohesion; so that they have high strength in order to be suitable for a given application. There are various types of sintering, i.e. gas-phase sintering (ZnO), liquid-phase sintering

(ceramic white wares and some refractories) and solid state sintering that consists of a host of ceramic materials with good mechanical and other properties of importance. Here, only the liquid-phase sintering will be discussed. The macroscopic driving force for sintering is the reduction of 'excess energy' associated with the surfaces is the main driving force. This reduction can take place mainly by two processes: total surface area is decreased by increasing the particle sizes leading to a 'coarsening stage', and solid/ vapor interfaces are eliminated with the creation of 'grain boundaries', which is followed by the 'grain growth' leading to 'densification or sintering'.

Actually, in a given ceramic material undergoing sintering, the above two processes are in 'competition'. However, the main goal is to reduce the former and optimize the latter, i.e. the densification. At a given sintering temperature, if the atomic processes leading to densification dominate, the pores start getting smaller and eventually disappear with sintering time, and the sintered compact shrink in size. However, if the atomic processes are such that the 'coarsening' is faster, both the pores and grains coarsen and get larger with time [6]. This obviously excludes the other processes like evaporation-condensation, and viscous and creep flow in sintering. Now, it is important to know which of the above diffusion processes is operative in the sintering mechanism. This is described in terms of a 'model' that is relevant for the sintering of nano particles of SiC in the along with the role of dopants in creating the necessary driving force for full densification. It has to be remembered that when the particles are small, say in the nano range, the total surface area is very large, which consequently gives a high surface energy for these nano particles. Hence, the driving force for reducing this surface is large enough to drive the sintering process to be completed faster. Therefore, it is prudent to go ahead to full densification by the preparation of the nano particles of SiC, which is otherwise difficult for a covalent solid like SiC [7]. The role of different additives and the dependence of different atmospheres of sintering have all been delineated in order to elucidate the problem of sintering further. This should help the readers to understand the mechanism of solid state sintering through 'practical' data on an actual material like SiC due to its characteristic properties [8].

The long-term oxidation behavior in air of pressure less liquid-phase-sintered SiC [9] was investigated as a function of the sintering-additive content (a mixture of  $Y_2O_3$  and  $Al_2O_3$  in the 3:5 molar ratio) at oxidizing temperatures in the interval 1100–1300 °C. It is shown that oxidation under these mild conditions is always passive, and with formation

of protective oxide scales. However, the oxidation kinetics cannot be described appropriately by the parabolic-rate law. Silicon carbide (SiC) is a highly covalent bonded compound which decomposes at 2730 °C. Therefore, it is difficult to densify SiC without sintering additives. Densification is achieved by solid-state sintering or liquid-phase sintering. Since the demonstration of successful densification of SiC with a toughened microstructure, interest in liquid-phase sintered (LPS)-SiC has grown continuously because LPS-SiC ceramics have better mechanical properties than solid-state sintered SiC ceramics [10].

There has been an increasing demand for high performance materials, which can withstand severe conditions such as abrasion, high temperature, pressure and atmosphere in various applications as follows: High temperature heat engines, nuclear fusion reactors, chemical processing industry and aeronautical and space industries. Therefore, the final products are cost-competitive besides offering the advantages of superior technical performance over the other materials [17-20].

In this investigation, the central goal is to examine the possibility of consolidating the covalent material with a reduced amount of additive, in the hope that materials with higher creep resistance can be produced. This is an indication that high-density silicon carbide could be produced by using mixed oxide as additive at lower cost. The sintering additives showed that  $Al_2O_3$ , and  $Y_2O_3$  are candidates because they do not react with SiC. The effectiveness of binary oxide systems can be explained by the effects of each oxide. In addition, the experimental observations were expanded to many other one component, binary and ternary systems for solid-state or liquid phase-assisted sintering, which were compared with previous research results [21-22].

## 2- Materials and methods:-

### 2.1. Materials

The starting material is the nano SiC that prepared from rice husk ash. Pure yttrium and nano alumina were used as sintering aides for the synthesized nano silicon carbide.

### 2.2. Equipments

SFM-1Disk-Top Planetary Ball Miller was used for milling the starting samples for 6 hour before pyrolysis. GSL 1600X furnace was used for the pyrolysis of rice husk ash at 1550°C. The carbon content was also confirmed and determined by Carbon and Sulphur detector, Model ELTRA 2000 MADE IN GERMANY. X-ray diffractometer type Philips, model Bruker D8 Advance-Germany, Target Cu K $\alpha$ , with secondary Monochromators, potential V

= 40 kV, current intensity A = 40 mA, and Ni filter was used to determine the mineralogical composition of the starting and synthesized materials. The patterns were recorded in  $2\theta$  range from  $20^\circ$  to  $70^\circ$ . The morphology and the particle size of the synthesized materials were examined by Transmission Electron Microscopy (TEM, JOEL JSM-1230). The obtained powders were pressed in a steel die mold iso-statically densified under 300 MPa. Microstructure of the sintered samples (Fracture surface) was examined by scanning electron microscope type (JEOL JXA-840A) after coating with a thin layer of gold under vacuum.

### 2.3. Methods

#### 2.3.1. Preparation of different samples of Nano SiC from rice husk

Two different starting SiC samples have been prepared: the first is rice-husk ash samples course (fired husk as is), the second sample is rice husk ball milled for 6 hours samples in 1Disk-Top Planetary Ball Miller. The two samples have been fired at  $1550^\circ\text{C}$  in vacuum.

Then the resulted nano SiC phases and the diffraction images detected by X-ray diffractometer, and the particle sizes measured from TEM images.

#### 2.3.2. Sintering of the produced Nano SiC

The sintering process was done on the two prepared nano SiC samples. The sintering mixture consist of nano SiC 90.00 wt%, nano yttrium of 3.00 wt. % and 7.00 wt% nano alumina as a sintering aides in an alcoholic solution (methanol). The sintering mixtures were homogenized in a plastic bottle by porcelain ball for 24 h, respectively. Then drying the mixture to evaporate methanol alcohol they were granulated and compacted. The obtained materials contained the samples were uni-axial pressed in a steel die mold iso-statically densified under 300 MP<sub>a</sub> by (SEIDNR-Riedlinger type, Germany).

The two resulted compacted for the nano SiC samples were then subjected to firing for 2 hour under vacuum and heating rate was  $10^\circ\text{C}/\text{min}$ ; at different temperatures ( $1750^\circ\text{C}$ ,  $1850^\circ\text{C}$ ,  $2000^\circ\text{C}$ , and  $2050^\circ\text{C}$ ) to reach the optimum sintering temperature. The cooling rate was  $20^\circ\text{C}/\text{min}$ .

#### 2.3.3. Characterization of Sintered Nano silicon carbide

The microstructure was observed by scanning electron microscopy (SEM). Sintered density was measured by the Archimedes' method. The theoretical densities of the specimens were calculated according to the rule of mixtures. Phase identification of the samples was performed by XRD.

- Microstructure of the sintered samples (Fracture surface) was examined by scanning electron microscope type (JEOL JXA-840A) after coating with a thin layer of gold under vacuum.

- The Archimedes displacement method was used for the determination of the bulk density and apparent porosity. This principle based on soaking the tested samples in kerosene under continues suction for 2hr. The saturated samples were weighted ( $W_s$ ) in air and then weighted after immersed in kerosene ( $W_i$ ). The dry weight was taken after leaving the samples overnight at  $110^\circ\text{C}$ . The bulk density (BD) and the apparent porosity (AP) of the test samples were calculated according to the following equations (1 and 2):

$$AP = \frac{W_s - W_d}{W_s - W_i} \times \gamma$$

$$BD = \frac{W_d}{W_s - W_i} \times \gamma$$

$W_d$ : weight of dry sample in air.

$W_s$  : weight of saturated sample in air.

$W_i$  : weight of immersed sample in kerosene.

$\gamma$  : specific gravity of kerosene.

Scanning electron microscope (SEM) investigations were done in order to examine the microstructure of sintered SiC parts. The samples were pyrolyzed in an alumina furnace at  $1500^\circ\text{C}$  for 2 h in air to reveal the grain boundaries.

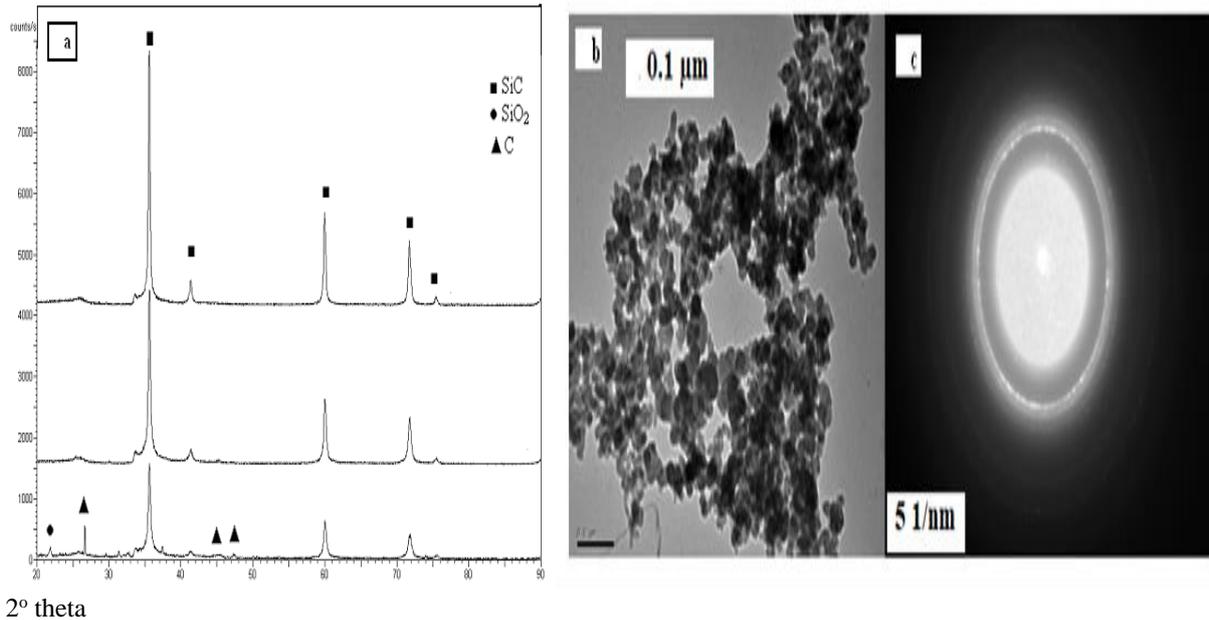
### 3. Results and discussion

As shown in Figure 1 well crystalline 20 nm SiC was obtained at  $1550^\circ\text{C}$  (TEM& diffraction), no characterizing peaks of silica and carbon were detected. During the pyrolysis at  $1550^\circ\text{C}$ , all the silica is converted to SiC, higher recrystallization and coagulation of SiC particles are occurred.

The sintering of the prepared nano SiC powders always needs sintering aids because of low sinterability of the covalent SiC itself. Depending on the kinds of the doped sintering aids, SiC may be sintered to high density by either a solid-state or liquid-phase sintering mechanism. Sintering of SiC (Fig 1b and Fig1c) is regarded to take place by solid diffusion process [11]. This technology usually requires sintering temperatures higher than  $2100^\circ\text{C}$  and easily results in exaggerated grain growth which is detrimental to mechanical properties. In contrast, the liquid-phase sintering process can be carried out at temperatures between 1850 and  $2000^\circ\text{C}$ . A wide variety of rare-earth oxides, usually in combination with  $\text{Al}_2\text{O}_3$  and/or boron compounds, could be used as sintering aids for SiC to accomplish a liquid-phase sintering process in which the oxides forms a liquid

phase at a temperature as low as 2000 °C. It was recently shown that SiC powder with the addition of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> could be sintered at lower

temperatures (about 1850–2050 °C) than the conventional sintered boron- and carbon-doped SiC [12–14].



**Figure 1:** a-XRD patterns of SiC fired at 1550 °C in vacuum (Top), b- TEM and diffraction (bottom); c- the bare is 200 nano meters.

**Table 1:** The bulk density and apparent porosity of all sintered SiC bodies under various conditions

Porosity at 2050°C	Porosity at 1950°C	Porosity at 1850°C	Porosity at 1750°C	Nano SiC sintering mixture
bulk density, g/cm <sup>3</sup>				
57%	54%	59%	63%	Nano SiC powder (No sintering additives)
1.3	1.5	1.15	1.34	
41.5%	34.5%	40%	43%	Nano SiC powder & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>
2	2.5	1.7	1.8	
12%	7%	11%	15%	Nano SiC powder prepared from ball milled rice husk ash & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>
2.93	3	2.95	2.91	
18%	13%	16%	46%	Nano SiC powder prepared from ball milled rice husk & stearic acid 1 wt% ash and & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>
2.9	2.915	2.915	2.62	

**Table 2: Relative density of the sintered SiC samples in relationship with SiC sources, additives and sintering temperature.**

Relative density, %				Nano SiC mixtures
2050°C	1950°C	1850°C	1750°C	
43%	46%	41%	37%	Pure nano SiC powder
58.5%	65.5%	60%	57%	Nano SiC powder & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>
88%	93%	89%	85%	Nano SiC powder prepared from ball milled rice husk ash & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>
82%	87%	84%	83%	Nano SiC powder prepared from ball milled rice husk ash & stearic acid 1 wt% & 3 wt% Y <sub>2</sub> O <sub>3</sub> & 7wt% Al <sub>2</sub> O <sub>3</sub>

From Table 2, it is indicated that the relative density of sintered SiC body obtained from pure nano SiC pyrolyzed at 1750°C is only 37%. By increasing the sintering temperature, the relative density has been increased and reached to the maximum value (46 %) at 1950°C. In addition, large shrinkage and cracks were observed in all these kind of samples. So such samples need extra sintering studies.

On the other hand, the relative density of sintered nano SiC prepared after milling of the rice husk for 6h with or without addition of 1% stearic acid has been increased as compared with those prepared from pure and impure un-milled ones. The relative density of the sintered SiC prepared from ball milled rice husk ash with or without stearic acid increased by increasing the sintering temperature reaching the maximum values 87 and 92% at 1950°C, respectively.

The most promising additives for liquid phase sintering are metallic or oxide forms, such as aluminum, mixtures of alumina and yttria [15–19]. However, a problem here is that the oxides react with SiC, volatilizing SiO, Al<sub>2</sub>O<sub>3</sub> and CO gases. Table 3 illustrates the relative density of sintered SiC samples at different firing temperature. It can be seen that the higher sintered bodies were obtained at 1950°C. The oxides additives played a crucial role in the formation of liquid phase between SiC particles so the relative density was consequently increased. In all samples, the relative density is generally increased after firing up to 1950 °C with a maximum relative density and then decreased after firing at 2050 °C. This phenomenon seems to be due to increasing the volatilizing of SiO and CO.

The microstructures of pure and impure (without or with milling) silicon carbide ceramics sintered at 1950 °C with two magnifications are shown in Figs. 2, 3, and 4, respectively. Generally, the use of relatively low sintering temperature, allows sub-micron grain size to be retained with moderate grain growth.

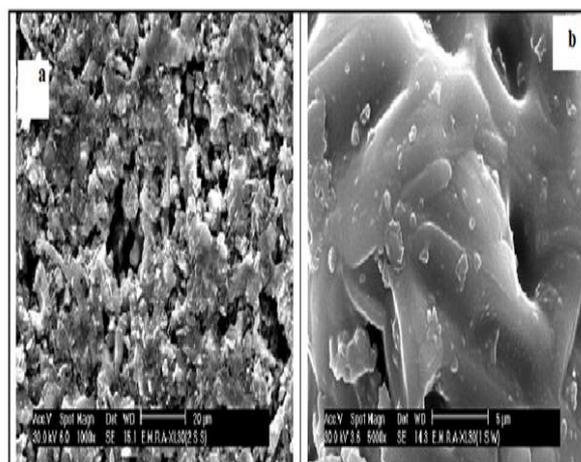
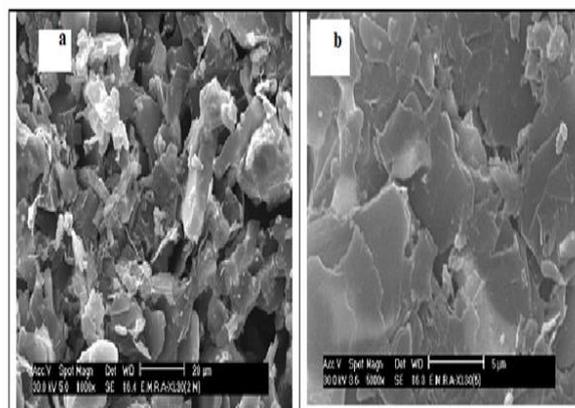


Figure 2: SEM photomicrographs of pure SiC sintered at 1950°C: a- of 20 μm, b- of 5 μm.

Figure 3: SEM photomicrographs of SiC containing 3 wt% Y<sub>2</sub>O<sub>3</sub> & 7wt% Al<sub>2</sub>O<sub>3</sub> sintered at 1950°C: a- of 20 μm, b- of 5 μm.

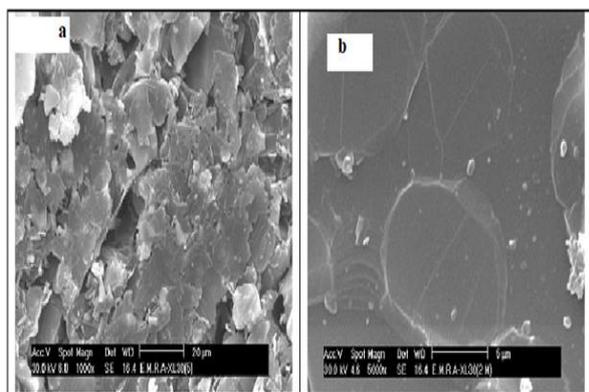


Figure 4: SEM photomicrographs of SiC prepared from ball milled rice husk ash & 3 wt%  $Y_2O_3$  & 7wt%  $Al_2O_3$ , sintered at 1950°C: a- of 20  $\mu m$ , b- of 5  $\mu m$ .

Porous microstructures are obtained for the sintered SiC prepared from rice husk without milling (Figs 2-3). The addition of 3 wt%  $Y_2O_3$  & 7wt%  $Al_2O_3$  increases the sinterability and consequently decreases the porosity (Fig. 3a, 3b). Relatively dense microstructure with smaller grain size is obtained for sintered SiC prepared from ball milled rice husk ash with presence of 3 wt%  $Y_2O_3$  & 7wt%  $Al_2O_3$ .

It is also indicated from the micrographs (Fig 4a and 4b) that the samples containing  $Al_2O_3$ – $Y_2O_3$  additives exhibited weak interface resulting from thermal expansion mismatch between liquid phase and matrix on cooling after sintering [20-21] and the sintering additives  $Al_2O_3$ – $Y_2O_3$  seem to reside at the grain boundaries and constitute the grain boundary phases. It is well known that [22-26] the difference in the grain boundary chemistry (oxide versus non-oxide) would lead to the differences in the thermo elastic properties of the grain boundary phases themselves and the chemical bonding strength between the SiC grains and its grain boundary phases, which results in different interfacial strength. A strong interface would hinder the occurrence of interfacial de-bonding, which is a basic premise for the crack bridging process.

#### 4- Conclusions

The present set of experiments has demonstrated a carbothermal reduction of two rice husk ash could be greatly enhanced by ball milling prior to the reaction. The simultaneous carbothermal reduction of rice husk ash could be greatly enhanced by high-energy ball milling prior to the reaction. The formation of nano SiC at the lower temperature 1500 °C is considered as a very economic process. The particle sizes of the obtained nano SiC from ball milled starting materials were smaller than those prepared with those grinded manually. This is followed by sintering of nano SiC that prepared from the two different starting techniques. The liquid phase sintering conditions of nano SiC powder have

been optimized sintering: The optimum sintering temperature was 1950 °C and the more efficient in sintering was ball milled rice husk ash without any surfactant. In this investigation, the central goal is to examine the possibility of consolidating the covalent material with a reduced amount of additive, in the hope that materials with higher creep resistance can be produced. These results suggest that the mechanical properties of liquid phase sintered SiC can be improved by the optimization of inter granular phase. This is an indication that high-density silicon carbide could be produced by using mixed oxide as additive at lower cost. The sintering additives showed that  $Al_2O_3$ , and  $Y_2O_3$  are candidates because they do not react with SiC. The effectiveness of binary oxide systems can be explained by the effects of each oxide. In addition, the experimental observations were expanded to many other one component, binary and ternary systems for solid-state or liquid phase-assisted sintering, which were compared with previous research results.

#### Conflicts of interest

Authors Ensure and declared that there is no Conflict of interest included in our manuscript here.

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#### عنوان البحث

تحضير كربيد السليكون الصلب بطريقة التصليب السائل بالتمركز والمحضر من حرق بقايا قشر الأرز المصري

المؤلفون : محمد عبد الجواد زايد ، محمود فرج زورة ،<sup>2</sup>مصطفى رجب علي  
إكلية العلوم جامعة القاهرة- 2- مركز التميز العلمي بالمركز القومي للبحوث وحدة السيراميك

#### ملخص البحث

يهدف هذا البحث الي تحضير نانو كربيد السليكون في صورة صلبة والمحضر سلفاً من حرق بقايا قشر الأرز بالحرارة في صورة نانوية وذلك للاستفادة من تلك النفايات البيئية وحماية البيئة المصرية منها. وقد تم في هذا البحث معالجة عينتين من قشر الأرز أحدهما محضر بالطحن اليدوي والأخرى آلياً في صورة حجم النانو. ووجد ان أفضل درجة حرارة لمعاملة تلك العينات oC1550 لتصل للصورة البلورية. وقد تم تجريب معالجة العينات حرارياً تحت جو مفرغ من الهواء أو جو خامل. وقد تم أيضاً خلط تلك العينات بنسب مختلفة من أكسيد الأتريوم (3% or 7%) قبل المعالجة الحرارية للمساعدة في تصلبها وهي سالة بالمعالجة الحرارية. وهذا الخلط لعب دور اساسي في انقاص نفاذية الخليط ومن ثم ارتفاع كثافته. وتم معالجة عينات الخليط حرارياً عند درجات حرارة وظروف مختلفة ووجد أن أفضلها درجة الحرارة oC1950 وأفضل نسبة من أكسيد الأتريوم هي 3%. وينصح أهمية هذا البحث في استخدامات كربيد السليكون المتصلد في العديد من التطبيقات الصناعية مثل صناعة السيراميك والمواد ذات التطبيقات الإلكترونية والطبية والبيوتكنولوجي. وكذلك تحويل مخلفات البيئة التي تشكل مشكلة في مصر وهي بقايا قش الأرز الي مواد مفيدة طبياً وصناعياً.