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ARTICLE

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WASTE TO WEALTH – RICE HUSK TO SILICON NITRIDE-BASED RADOME MATERIALS

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The prime goal of the 21st century is social, economic and environmental sustainability. Sustainability can be achieved only by renewable resource management. The use of the waste resource of rice husk to generate an engineered material of silicon nitride for aerospace applications is a stepping stone toward renewable resource management. This paper described the transformation of rice husk to amorphous silica followed by silicon nitride through carbothermal reduction nitridation. The formation of silica and silicon nitride was confirmed through powder X-ray diffraction analyses and infrared spectroscopy. The electromagnetic measurements confirmed the radar transmission properties of silicon nitride indicating the suitability of fabricated rice husk-converted silicon nitride as high-temperature ceramic radome material.

Keywords: *Rise husk, silicon nitride, sustainability, aerospace materials, dielectric permittivity, electromagnetic window, radar*

Introduction

Waste-to-Wealth stands for transforming waste from a platform of exhausted form to a valuable added level. Waste management system in a circular economy turns around the scope of recycling, reducing and reuse of recycled materials. One such value-added material is high-temperature ceramic radome. Radome is a protective enclosure for radar antennas that is mounted on airborne or ground systems. Radome should be electromagnetically transparent and mechanically and environmentally stable. The accurate designing of the radome depends on cautious choice of materials properties and optimization of the thickness of the radome wall. Radome provides environmental shielding for the antenna under harsh climatic conditions along with erosion and abrasion resistance, moisture and temperature tolerance, supports aerodynamic load, increases the lifespan of the antenna and other components, reduces the overall maintenance cost of the antenna, provides an optimized environment for the operation of the antenna with nominal signal degradation[1,2].

The aviation radars work primarily in the microwave frequency ranges of 4-18 GHz. To obtain effective radar communication, the EM enclosure should allow preferably 100% transmission. The electromagnetic transparency is determined by low dielectric constant (ε) and low loss tangent (tan δ) of the material. The composite radome exhibits broadband transmission between 8 and 20 GHz, which is associated with excellent mechanical and electromagnetic properties[3]. However, they have the restriction of functionality on operational temperatures beyond 250ºC[4]. High-speed military aircraft require high-

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temperature ceramic radome with superior mechanical and physical properties in the temperature range of 500–800ºC with high tolerance in tougher atmospheric conditions. Fused silica is an important material for high-temperature applications because of its low density and good resistance to thermal shock up to 1000ºC. Additionally, it has excellent dielectric properties of low dielectric constant (ε ~ 3.06–3.32), low loss tangent (tan δ ~ 0.00053–0.0065) with good stability of these parameters with temperature variation (between 25ºC and 1000ºC)[5,6]. However, fused silica exhibits a low mechanical strength of 65-70 MPa. Silicon nitride, on the other hand, is a material of interest due to its outstanding combination of properties such as a tailorable density in the range of 1.8 to 3.0 g/cm^3, dielectric permittivity ε ~ 4-6, and loss tangent tan δ ~ 0.001-0.006 accompanied by high mechanical strength up to 1300ºC, corrosion and wear resistance, high hardness, low density and high chemical stability[7-11].

Rice husks (RH) are the hard protective covering of the rice grains which are by-products of the paddy and separated from the rice grains during the milling process. RH is an agricultural waste material that is mostly destroyed by burning in open space causing huge environmental pollution. Several efforts have been made to utilize RH beyond cattle feeding and landfilling, such as bioenergy as an alternative fuel and fertilizers[12-14]. RH accounts for 20% of the total rice production of the world which is estimated at 700 million tons. India is the second largest in the production of rice creating approximately 30 million tonnes of rice husk per year. RH is mainly composed of 50% cellulose, 30% lignin group and 20% silica. Combustion of RH in air produces rice husk ash which contains 85-98% silica[15].

This paper describes the journey of waste rice husk to an important aerospace material, silicon nitride, a potential candidate as a high-temperature radome material for strategic applications. The use of waste resources to create engineered silicon nitride is a precious example of sustainability. Rice husks are low-cost renewable resources that has been used to generate silicon nitride. The series of chemical procedures involved in the development of silicon nitride-based radome materials from rice husk and the properties of the synthesized silicon nitride are described in this paper.

Experimental

Methodology: **Preconditioning of the Rice Husk:** Initially, rice husk was washed thoroughly several times to remove all the dirt present. The washed rice husk was dried in the oven at 70 °C. After the RH was dried completely, acid leaching was carried out using concentrated hydrochloric acid. The dry RH was boiled with concentrated hydrochloric acid at 70-80 °C for several hours. The acid leaching procedure was done twice followed by washing the RH with distilled water to remove the excess water. Final drying was done in the hot air oven for 48 to 72 hours at 200 °C.

Rice Husk to Silica: The dried RH was taken to 700 °C in the air for several hours to convert RH to amorphous silica as a white powder.

Silica to Silicon Nitride: Silica obtained from rice husk was converted to silicon nitride by carbothermal reduction nitridation[16,17]. In this process, amorphous silica was mixed with carbon black in the molar ratio of 1:5. The mixed powder was pressed in the pellet form to have an effective reduction nitridation reaction. Carbothermal reduction nitridation was carried out under constant flow of nitrogen gas at two temperature regions of heating, 1000 °C for a short period followed by 1500 °C for several hours. The pellets crashed after the reaction and the powder was heated at 800 °C in an air atmosphere for de-carbonization reaction of excess carbon. The resultant material was silicon nitride, the formation of which was explained by the following chemical reactions.

$$3SiO_2 + 2N_2 + 6C \rightarrow Si_3N_4 + 6CO$$

Silicon nitride powder was mixed with silicon powder in the ball mill for several hours in the presence of ethanol as a mixing medium. After the mixture was dried, the pellet was fabricated using the cold uniaxial press. The pressed pellet was reaction bonded to compacted silicon nitride disc under the nitrogen atmosphere at temperatures up to 1500 °C for several hours.

Characterization of Materials: The phase determination was carried out on powder *x*-ray diffraction on M/s. Bruker D8 Advance *x*-ray diffractometer with Ni-filtered Cu $K\alpha$ radiation. The FTIR spectra were recorded on the Perkin Elmer FTIR spectrometer. The dielectric constant (ε) and dielectric loss (tan δ) were measured in the frequency range, 4-22 GHz using a vector network analyzer based on circular cavity method.

Results and Discussion

Silica is silicon dioxide that consists of silicon atoms tetrahedrally bonded with four oxygen atoms. Further, each oxygen atom bonded with four silicon atoms and forms a network of –O–Si–O–Si–O– and gives rise to a large silica matrix. It exists in two primary forms: amorphous and crystalline. Silica has pores ranging from 5 to 3000 Å, which decides the stabilization of the polymorph as amorphous or crystalline[18]. In this study, the FTIR and XRD together confirm the formation of silica from rice husk. Fig. 1 and 2 show the FTIR spectra and XRD pattern of silica obtained from the heat treatment of rice husk. The notable absorption peaks at 1080 cm^{-1} and 813 cm^{-1} feature O-Si-O stretching and bending vibrations, confirming the formation of silica[19]. Further, the absence of a crystalline peak in the XRD pattern confirms the formation of amorphous silica during the heat treatment of rice husk.

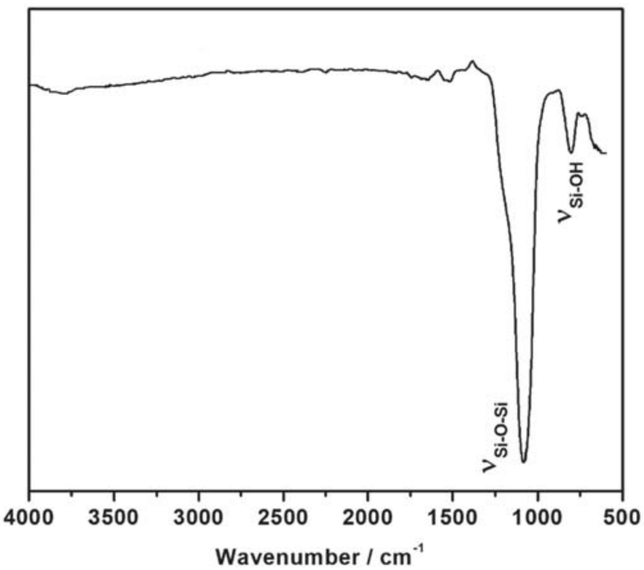

Fig. 1: FT-IR spectrum of silica obtained from heat treatment of rise-husk.

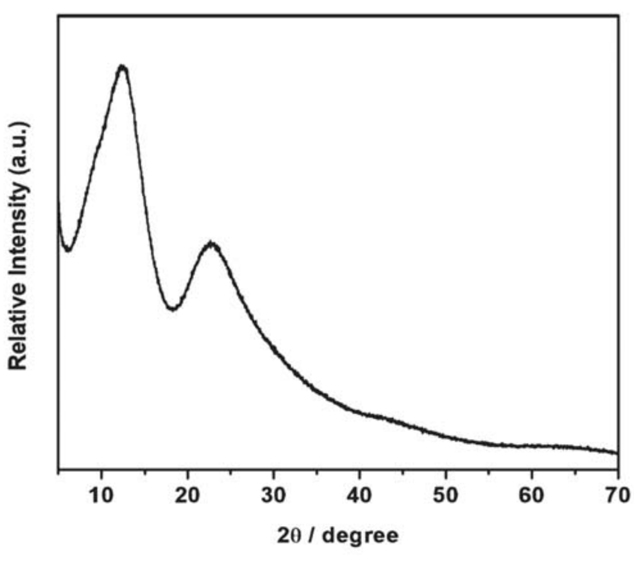

Fig. 2: XRD patterns of silica obtained from heat treatment of rise-husk.

The carbothermal reduction nitridation converts silica obtained from rice husk to silicon nitride. Silicon nitride is formed as a layer of white felt in a loosely bonded pellet

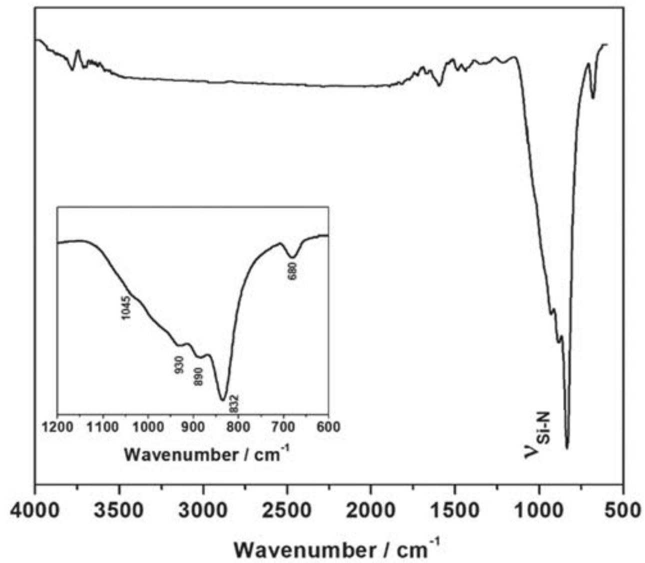

Fig. 3: FT-IR spectrum of silicon nitride obtained from carbothermal reduction nitridation of rice-husk silica. Inset shows enlarged image.

form. The sample is retrieved by tweezer and the unreacted carbon is burnt off by heat treatment in the air atmosphere. The carbon goes off as carbon dioxide leaving silicon nitride as residue. The formation of silicon nitride is identified using powder XRD and FTIR spectra. Fig. 4 shows the powder XRD pattern of silicon nitride obtained from rice husk-converted silica. The XRD pattern shows the presence of both α and β-phases of Si_3N_4.[11] All the lines of α-Si_3N_4 from 2θ of 5 to 70° are indexed as α-Si_3N_4 (JCPDS Card No. 76-1407) whereas the only prominent peaks of β-Si_3N_4 (JCPDS Card No. 82-698) was identified in the lower angle region. The broad bulge between 5 to 15° arises from the plastic sample holder of the XRD probe. The formation of Si_3N_4 is further confirmed by the FT-IR

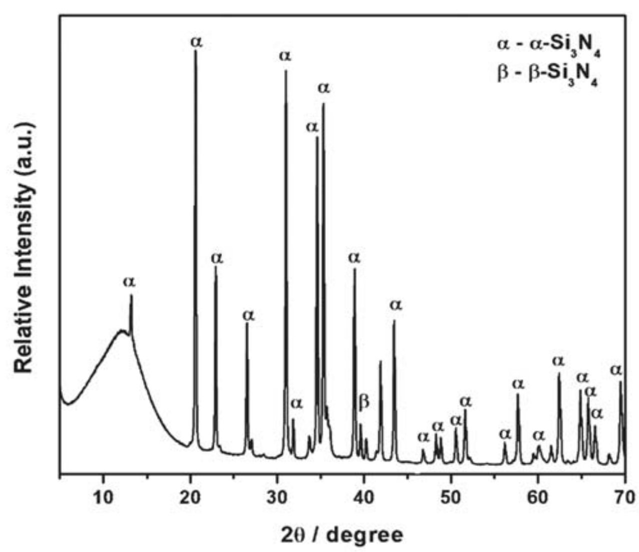

Fig. 4: XRD pattern of silicon nitride obtained from carbothermal reduction nitridation of rice-husk silica.

spectrum as shown in Fig. 3. The absorption peak ranging from 800 cm^{-1} to 1100 cm^{-1} is attributed to the Si-N-Si skeleton vibration of Si_3N_4. The peak at 680 cm^{-1} signifies the α-phase of Si_3N_4[20].

The morphology of the silicon nitride sample obtained from rice husk silica is examined by scanning electron microscopy (SEM). Fig. 5 shows the SEM image of a compact silicon nitride sample. The evenly distributed near spherical microstructure with grain size of 0.5-2 μm is observed from the micrograph.

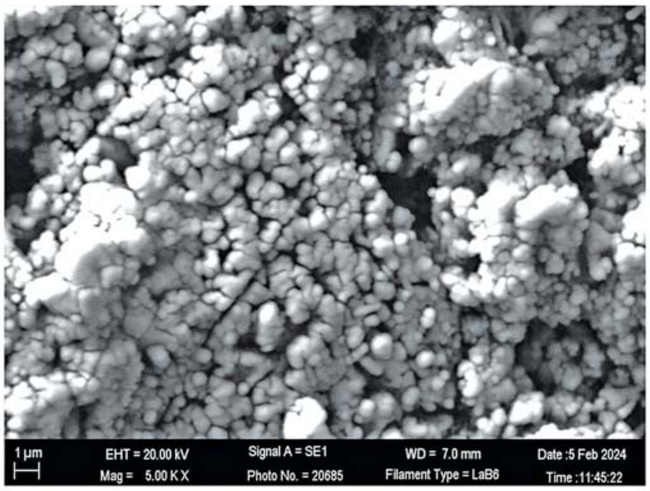

Fig. 5: SEM micrograph of silicon nitride compact obtained from rice-husk silica.

To understand the functionality of rice-husk converted Si_3N_4 as an electromagnetic (EM) transmission window, the dielectric permittivity and loss tangent analyses were carried out based on measured data. Fig. 6 shows the measured dielectric permittivity (ε) and loss tangent (tan δ) as a function of frequency. The dielectric permittivity (ε) increases from 2 to 5 in the frequency range of 4-8 GHz. The ε stabilizes above 8 GHz and remains almost

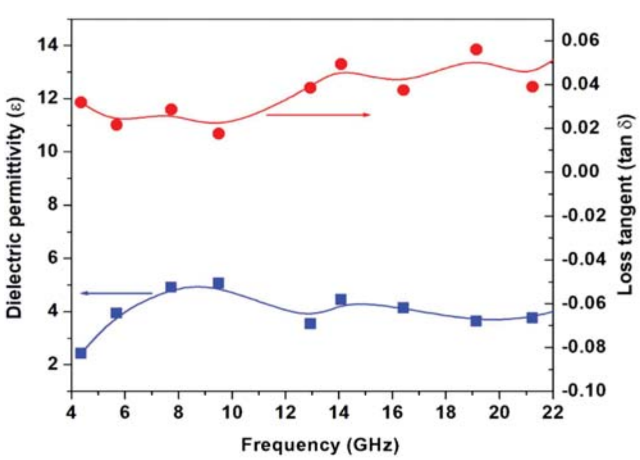

Fig. 6: Dielectric permittivity (å) and loss tangent (tan ä) as a function of frequency.

constant between 10-22 GHz. The loss tangent values vary between 0.02 and 0.05 in the entire frequency range of 4-22 GHz. The EM analyses indicate that Si_3N_4 obtained from rice husk is suitable for electromagnetic window application. A rigorous purification process will further improve the EM properties.

Conclusions

Rice husk is a promising source of silicon nitride, an aerospace material. The primary application of silicon nitride is high-temperature radome which is an enclosure for antenna. In terms of rice production, our country ranks second with an annual production of more than a hundred million tonnes of rice per year. Approximately 30 million tonnes is the amount of rice husks obtained each year. This paper highlights the conversion of rice husk to usable aerospace materials of silicon nitride. The systematic process development not only produces more efficient radome materials, also will reduce the import cost of the raw materials.

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