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Synthesis and characterization of nanoparticles of wurtzite aluminum nitride from various nut shells

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Nanoparticles of Aluminum Nitride (AlN) were synthesized from a thermal treatment of mixtures of aluminum oxide (Al_2O_3) and either shells of almond, cashew, coconuts, pistachio, or walnuts in a nitrogen atmosphere at temperatures in excess of 1450 °C. By selecting the appropriate ratios of each nutshell powder to Al_2O_3, it is demonstrated that stoichiometric aluminum nitride is produced via carbothermal reduction in nitrogen atmosphere. In addition, results show the formation of Al from Al_2O_3 before transformation to AlN. On the other hand, when Al was mixed with nutshell powder first, mixed phases of AlN and Al_4C_3 were formed before complete transformation to AlN. X-ray diffraction analysis, Raman scattering and Fourier Transform Infrared spectroscopy confirmed the wurtzite phase of aluminum nitride. Transmission electron microscopy indicated the formation of AlN nanoparticles. The formation of AlN from nutshells offers a simple route and avoids multiple-step processes involving carbon rich agents at elevated temperatures.

1. Introduction

Aluminum nitride belongs to a class of III-N materials and is a wide gap semiconductor with band gap between 6.01 and 6.05 eV at room temperature [1]. It exhibits both wurtzite and zinc-blende phases as do other III-N compounds and has many potential applications in microelectronics due to its relatively high thermal conductivity (70–210 W m^{-1} K^{-1} to 285 W m^{-1} K^{-1}) [2]. Additionally, it has unique physical properties including high electrical resistivity, low thermal expansion, resistance to erosion and corrosion, excellent thermal shock resistance and chemical stability in air up to 1380 °C with surface oxidation occurring at 780 °C. Moreover, epitaxially grown thin films of crystalline aluminum nitride are used for surface acoustic wave sensors (SAWs) deposited on silicon wafers because of AlN's piezoelectric properties [3]. Another important application for AlN is as an RF filter, also called a thin film bulk acoustic resonator (FBAR), which is widely used in mobile phones [4]. AlN in bulk form is synthesized by the carbothermal reduction of aluminum oxide in the presence of gaseous nitrogen or ammonia or by direct nitridation of aluminum. In order to get fully dense form, Y_2O_3 or CaO are required as additives during the hot pressing.

There are two types of agriculture waste, one containing silica and carbonaceous matter and the other containing mostly carbonaceous matter and no silica. Among the first type, the prominent ones are rice husk, wheat husk, corn husk, sorghum leaves and peanut shells. We have demonstrated in our previous work that these are useful in the synthesis of industrially important materials such as SiO_2, SiC, Si_3N_4, and zinc silicate by pyrolizing them in air, argon or in nitrogen atmospheres [5–11]. The second type of agriculture waste which contain only carbon matter and no silica are nutshells such as almond, walnuts, pistachio, coconuts, macadamia, and cashew. Billions of pounds of nutshells, which are currently discarded as waste products, are available if they can be harnessed in the synthesis of industrially important materials. Various tree nut shells are frequently used for organic composts, animal feedstock, or discarded as trash [13]. The present study demonstrates a more effective and practical way of utilizing tree nut shells by utilizing them as a carbon source in synthesizing AlN, that will have widespread industrial implications.

Recently, it was reported that mixed phases of SiC and Si_3N_4 can be produced by carbothermal reduction and nitridation of a mixture of silica and macadamia powder [12]. We demonstrated in our previous work that by adding ZnO to powder of wheat or rice husk, pure zinc silicate is produced with photo-luminescent properties [11]. In the present paper, we report on the formation of AlN by adding nanocrystalline powders of Al_2O_3 and Al to the

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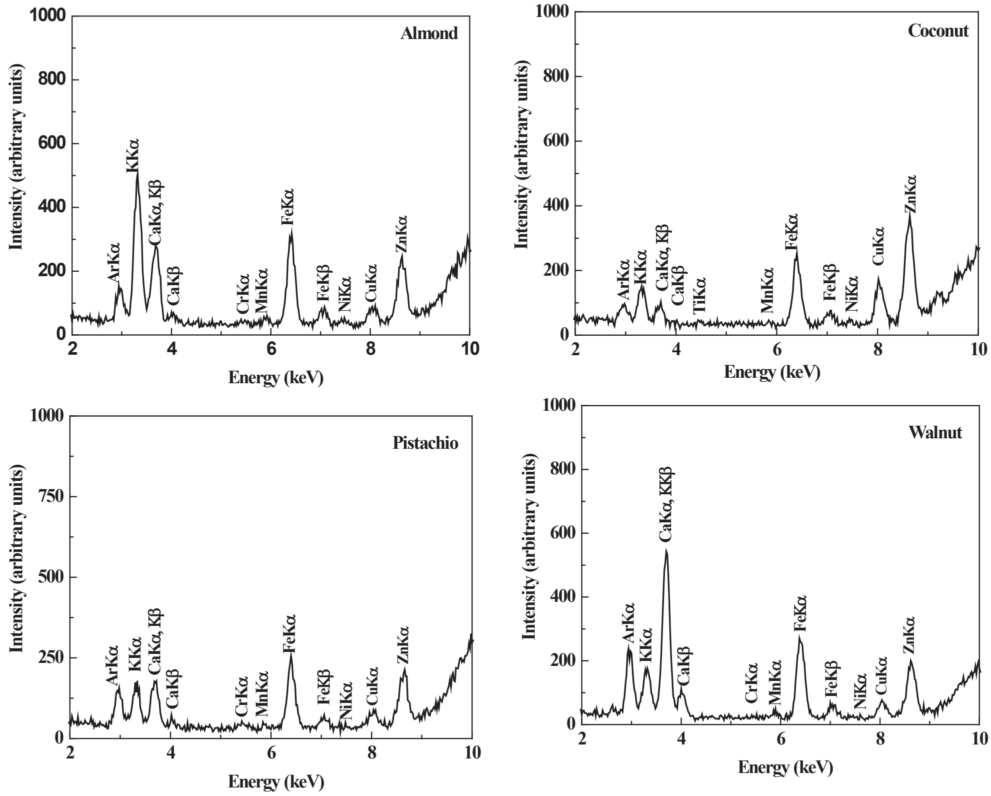

Fig. 1. Energy dispersive x-ray fluorescence of almond, coconut, macadamia, and walnut showing the presence of trace amounts of K, Ca, Cr, Mn, Fe, Ni, Cu, and Zn.

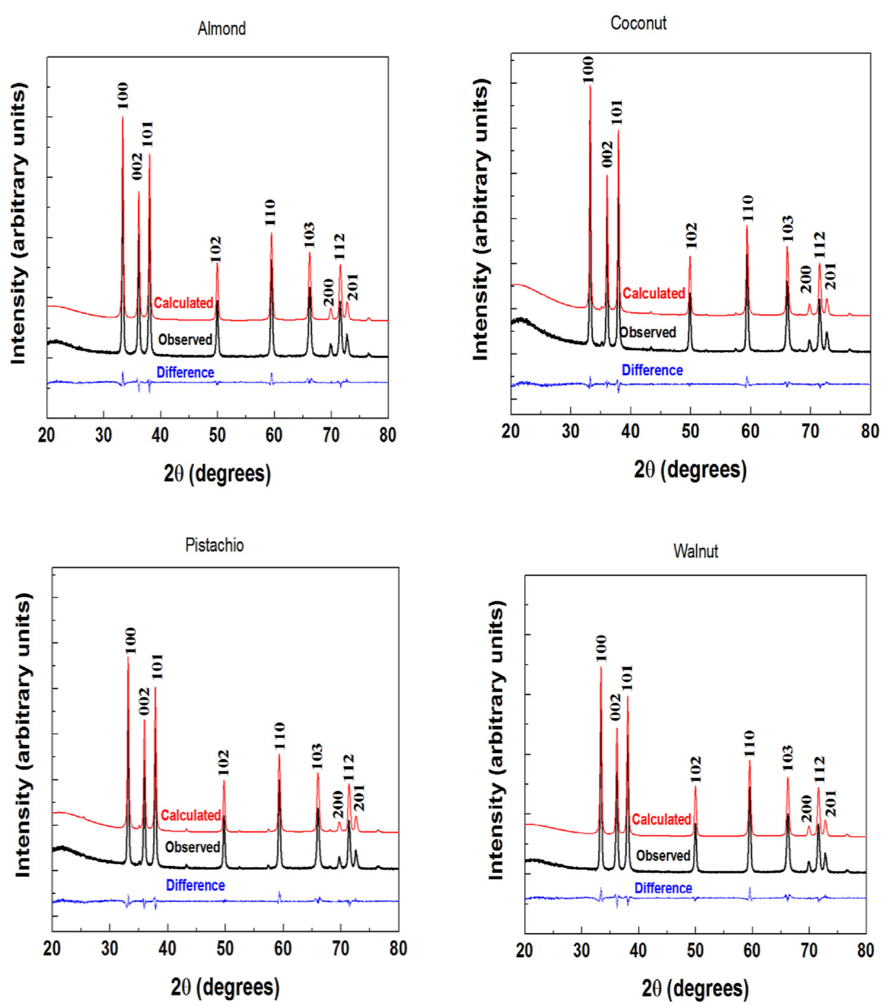

Fig. 2. X-ray diffraction scans of Al_2O_3 mixed with selected nut shell powders along with Rietveld whole profiles analysis of the diffraction patterns for the AlN sample derived from nut shells powder after pyrolising in a nitrogen atmosphere followed by treatment in air at 800 °C.