



PROCESSING AND CHARACTERIZATION OF HYBRID COMPOSITE THROUGH SOL- GEL TECHNIQUE

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Abstract: The scientific community is interested in processing materials that may be used in both biomedical and aeronautical applications. The complications involved in achieving this viability include customizing the processing parameters, material morphology, and constituent makeup. Assume that aircraft applications need material qualities such as high strength-to-weight ratio, corrosion resistance, heat resistance, and ductility. However, because it is fragile and requires a low strength-to-weight ratio, the same material with these characteristics is not appropriate for biological applications. In actuality, brittle materials are not mappable since they are acceptable for biological applications. The rationale for the introduction of this innovative concept is to realize a sustainable development objective for the clean energy system. Since the materials suitable for biomedical applications involve degradability and at the same time can be processed from bio-waste. But materials with these properties require further processing by dispersing various metals which can exhibit multidimensional properties. i.e., biodegradable materials lack a high strength-to-weight ratio, thermal resistance, and moisture resistance. To enhance these properties, combining the metals with biodegradable materials is of specific interest and hence the current work addresses this.

The extracted silica from rice husk is employed as a silica precursor in the current study, namely tetraethyl orthosilicate (TEOS), which is used to improve heat resistance. Although silica has great temperature resistance, it is fragile and hence cannot be utilized directly in aeronautical applications. This has been improved by dispersing precursors for nickel and titanium in nitride form to make it suitable for the sol-gel method. Further characterization will be done to see the powder's shape and the thermal analysis when the nitride forms are changed into oxide forms. Scanning Electron Microscopy (SEM) and TGA are used for morphology research and thermal analysis, respectively. Finally, a structure-property correlation will be created in order to investigate how the composition affects the powder's qualities.

IndexTerms - Hybrid Composite, Sol-zel, Aerospace, Biomedical, tetraethyl orthosilicate, Scanning electron microscopy.

I. INTRODUCTION

The two main energy sources that have drawn a lot of interest from scientists are sustainable and clean energy. As a result, turning bio waste into a useful product is one approach to this. It is widely accepted that sectors like aerospace, automotive, and biomedicine are responsible for the majority of material development. Researchers are still unable to create a class of materials that are suitable for all sectors on a single scale since these industries require similar qualities to one another. Instead, the present effort will concentrate on the viability of using processed biowaste as a raw material to manufacture parts appropriate for automotive and aerospace applications. It is exciting to learn that the scientific community has conducted a significant amount of study on a variety of materials, including composites, smart materials, intelligent materials, and functionally graded materials. Depending on the method of processing and the combinations of their constituent parts, these materials display a range of mechanical, tribological, physical, and thermal characteristics.

II COMPOSITES. Composites are a type of materials created when a matrix and reinforcement are joined with another material in a heterogeneous way. There are several metals, ceramics, polymers, and resins that may be used as the matrix and reinforcement. The advantages of the composites include the multidimensional properties i.e., for an instance, assume silica-based ceramic poses excellent thermal resistance but is brittle in nature and has lower tensile strength. To enhance the tensile strength and improve ductility, a combination of metal or polymer will be added heterogeneously to form the composite laminate. As a

result, the newly formed composite has both high thermal resistance (due to silica) and ultimate tensile strength (UTS) (due to metal or polymer). The synergistic reaction between the matrix and reinforcement plays a vital role in achieving good mechanical and tribological followed by thermal properties. This synergy can be achieved by choosing an appropriate matrix and reinforcement material.

The literature discusses many combinations in relation to it, including glass-reinforced carbon composites (GRCC), metal-matrix composites (MMC), ceramic matrix composites (CMC), polymer-matrix composites (PMC), etc. Along with being widely employed, these composites—whether in bulk or laminate form—have also shown limitations in a number of applications due to issues including poor interfacial strength, delamination, and stress concentration at the bonding joints. As a result, to enhance these properties new processing technologies have been adopted against the conventional techniques. Few technologies including hand layup (wet layup), wet chemistry approach, pultrusion, sieving, and chemical vapor deposition (CVD) have been widely used. However, though, it was identified that not a substantial enhancement in the properties has been observed. In contrast to the aforesaid techniques, we shall discourse on the advantages and limitations.

III. MATERIALS ARE USED IN THE CURRENT WORK. The following elements were employed as a predecessor in the current suggested study; their composition was recognized from the scientific literature and was found to be acceptable in earlier research. However, after much testing, we have determined that this composition is most appropriate and is proven to be in excellent accord with the hybrid structure we have chosen, thus it is addressed more below.

Name of the material	Chemical formula	Composition	Molecular weight
Tetra Ethyl Orthosilicate	$Si(C_2H_5O)_4$	6gm	208.33
Ethanol	C_2H_5OH	60ml	46.08
Ammonia	NH_3	3.5ml	17.003
Cetrimonium bromide	$C_{19}H_{42}BrN$	0.65mg	364.45
Deionized water	H_2O	25ml	18.01
Titanium nitride	TiN	0.25gm	61.874
Nickel(II)nitrate hexahydrate	$Ni(NO_3)_2 \cdot 6H_2O$	0.25gm	290.79
Total weight			1010 (approx.)

Table 1. Chemical composition of the material

IV SOL-GEL SYNTHESIS. The creation of Sol and subsequent transition into a gel, i.e., a colloidal system consisting of a liquid dispersion medium enclosed in a spatial grid formed by linked particles of the dispersed phase, is assumed in nanomaterials technology. Sol-gel synthesis is a very promising alternative approach for efficiently and cost-effectively producing nanocrystalline elemental, alloy, and composite powders. At significantly lower pressures and temperatures, nanocrystalline particles may be cemented.

The sol-gel technique is a flexible wet chemical method for producing ceramic and glass materials. This synthesis method includes converting a system from a colloidal liquid, known as sol, to a solid gel phase. The sol-gel process enables the preparation of ceramic or glass materials in a broad range of shapes, including ultra-fine or spherical powders, thin film coatings, ceramic fibres, microporous inorganic membranes, monolithics, and very porous aerogels. The figure below depicts an overview of the sol-gel process.

The low process temperature, the capacity to adjust the composition at the molecular level and the porosity to produce materials with large surface areas, and the uniformity of the finished product down to the atomic level are only a few benefits of this method. Moreover, it is possible to synthesize complex composition materials, to form higher purity products through the use of high purity reagents, and to provide coatings over complex geometries. The sol-gel process allows to obtain high quality films up to micron thickness, difficult to obtain using the physical deposition techniques. Moreover, it is possible to synthesize complex composition materials and to provide coatings over complex geometries. The starting materials used in the preparation of the sol are usually inorganic metal salts or metal organic compounds, that by hydrolysis and polycondensation reactions form the sol.

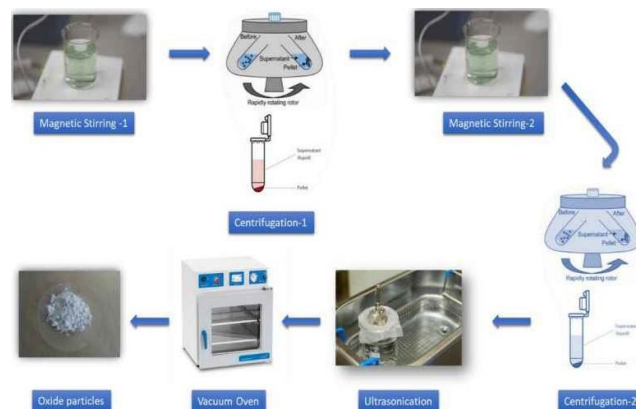


Fig.1 Sol-gel process

The sol can be processed further to produce ceramic materials in a variety of shapes. Both dip-coating and spin-coating are methods for creating thin films. Wet gel will develop when the sol is put into a mould. The gel is transformed into thick ceramic or glass materials by drying and heating. If the liquid in a wet gel is removed under a supercritical condition, a highly porous and extremely low-density aerogel material is obtained. As the viscosity of a sol is adjusted into a suitable viscosity range, ceramic fibres can be drawn from the sol. Ultra-fine and uniform ceramic powders are formed by precipitation, spray pyrolysis, or emulsion techniques.

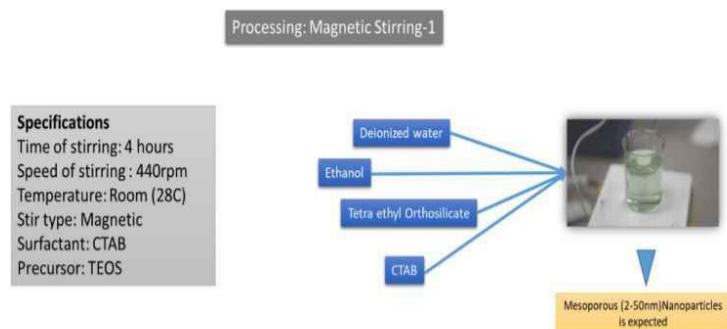


Fig 2. Stiffing of TEOS

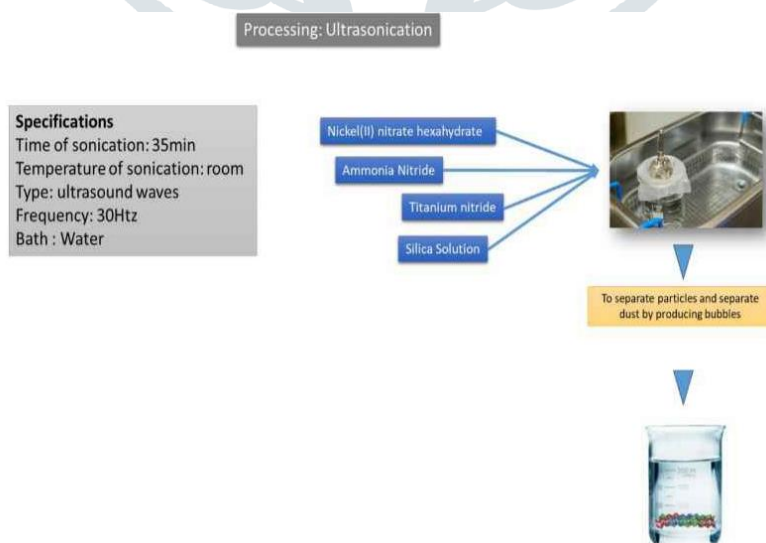


Fig3. Ultrasonication

The purpose of the sonication is to separate the particles from the nitrides by removing any dust from the procedure that is necessary. The gel is cooked in a vacuum heat oven following sonication to produce the appropriate powder form, as indicated in the picture below. In order to determine if the process has been tuned smoothly or not in order to achieve the desired properties, such as mesoporous, uniform porosity, and spherical in shape.



Fig 4. Process powder

V. RESULTS AND DISCUSSIONS. Following the fabrication of the hybrid composite nanopowders, the nanopowders were analysed for their microstructure and thermal studies to determine if the mechanical and thermal characteristics improved. SEM was used to explore the microstructures of the hybrid composite powders. These investigations helped us understand the form and size of the hybrid composite powders. Thermal studies were carried out using differential scanning calorimetry (DSC). The glass transition temperature and melting point were plotted against the heat flow rate in these results. The experiments additionally emphasis the phase transition from ductile to brittle or brittle to ductile. In addition, X-ray Diffraction is used to confirm the crystal structure of the system and the final percentages. In collaboration with XRD, FTIR is then used to determine if all of the compounds are present in the correct percentages.

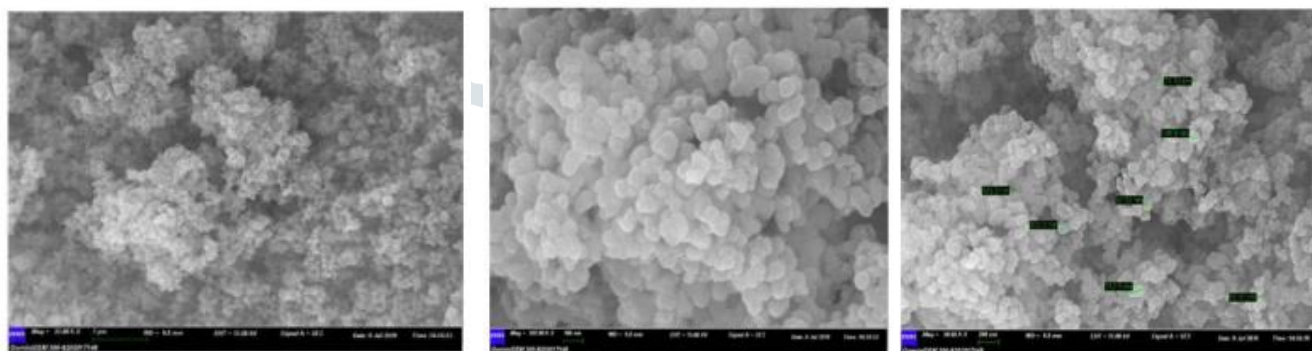


Fig 5. SEM of as received powder for 7500 rpm

The graphic below shows that there are agglomerations through the production of spherical particles, which suggests that silica formed as micelles and that the other metals were distributed equally at nanoscale sizes. However, metal particles developed as aggressive particles that made hydroxyl bonds with silica.

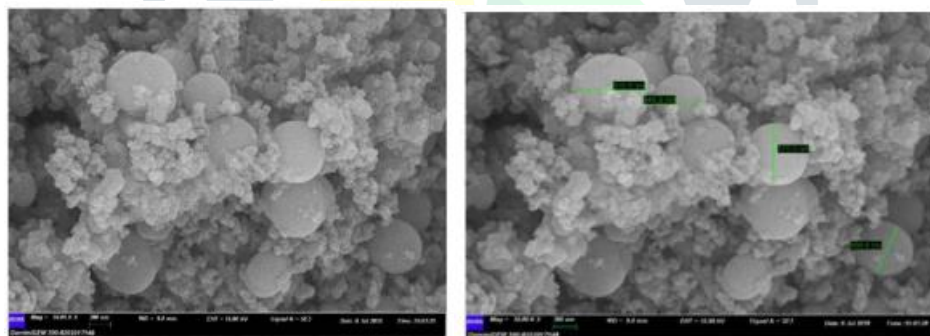


Fig. 6. SEM of as received powder for 7830 rpm

According to the preceding figure, the particle's minimum and maximum sizes are 488.6 nm and 676 nm, respectively. indicating that while the spherical diameters are attained with longer churning times, metallic agglomerations have developed. a nonuniformity brought on by faster stirring and higher centrifuge rpm..

The XRD results for nickel and titanium are shown below and were found to be identical to the COD database. The peaks have a NiTi stable configuration at the (110) plane with a monoclinic crystal structure, indicating that the system is in the martensitic phase at room temperature.

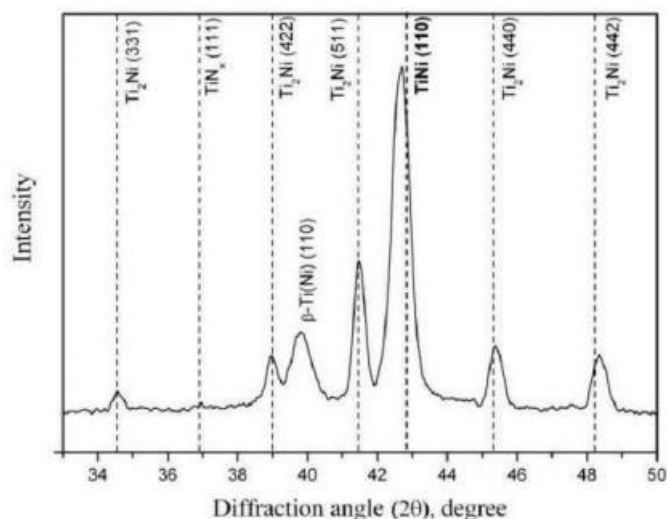


Fig. 7. XRD of Ni- Ti sample

A minor amount of the Ti-Ni phase can be linked to an increase in nickel content as a result of top layer evaporation following each compression plasma flow effect. This impact is mild, but because the melting point of nickel is lower than that of titanium, the nickel evaporates more fiercely. It allows for a reduction in the relative content of nickel in the changed layer. It validates the presence of diffraction peaks corresponding to the titanium phase that forms when the melt solidifies and is devoid of nickel atoms. Titanium solidified in a high-temperature β -phase in places with nickel atoms. The XRD of silica alone after being diffused from the whole powder via the sonication method is shown below. The X-ray Diffractometer (XRD) was used to characterize the produced silica nanoparticles using Cu Ka ($\lambda = 1.54$) as a radiation source. The figure shows the XRD patterns of silica nanoparticles produced from rice hulls. The powder diffraction pattern shows a large peak at $2\theta = 22^\circ$, indicating that the silica nanoparticles are amorphous. The XRD pattern further supports the lack of any organized crystalline structure.

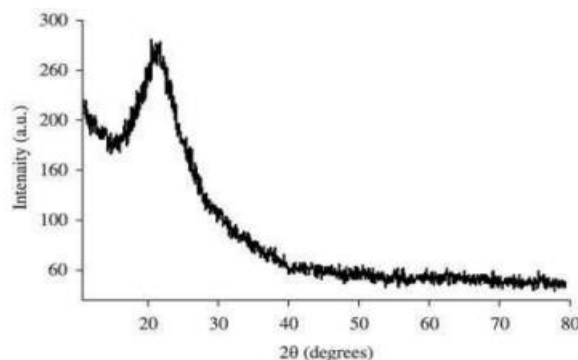


Fig. 8. XRD of Silica sample

As demonstrated in the picture below, the Fourier transform for silica discovered in the literature corresponds to the present test sample. The findings from infrared spectroscopy showed that ester cross-linkages in finished cotton may be investigated and quantified by measuring the ester band intensity and carbonyl band intensity ratio (ester/carboxylate). The carbonyl band intensity ratio (ester/ carboxylate) is a function of the average number of ester groups formed for each BTCA molecule, so it represents the effectiveness of this bonded BTCA molecule as a crosslinking agent. From the figure, it was observed that the carbonyl band in 1722 to 1735 cm^{-1} represent the carbonyl of ester while the band at 1577 to 1586 cm^{-1} represent the carbonyl of carboxylate. The figure below displays the separated Ni-Ti FTIR spectra which the spectrum of the surface of the chemically polished one, the one for the H2O2-oxidized one has some additional bands, e.g., two broad OH stretching bands at 3352 and 3233 cm^{-1} deriving from Ti—OH groups and the band at 1625 cm^{-1} corresponding to the bending mode of chemisorbed water.

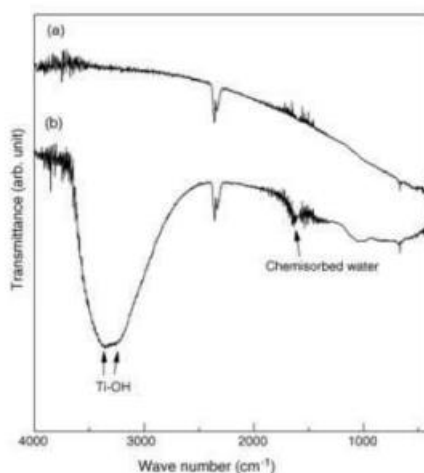


Fig. 9. FTIR of Silica and Ni-Ti sample

By monitoring the weight change that occurs while a sample is heated at a consistent rate, the TGA analytical technique may be used to determine a material's thermal stability and the percentage of volatile components. Mass changes often correspond to chemical reactions. A typical example is drying, which can be thought of as a brief initial drop at the beginning of heating that doesn't appear to be related to any chemical processes.

Evaporation/sublimation Evaporation/sublimation may also appear on the plot depending on the material to be analyzed. Multistage decomposition is also common and shows a step-like pattern. In some cases, these steps may blend during dynamic TGA, necessitating either far slower heating rates or stepwise methods like quasistatic TGA. Note that TGA itself may not be sufficient to identify the decomposition products; chemical testing of the sample after TGA analysis is often required to ascertain the identities of suspected decomposition products. TGA itself does not identify substances; other methods such as chemical testing or differential calorimetry must be performed alongside TGA to verify the identity of products.

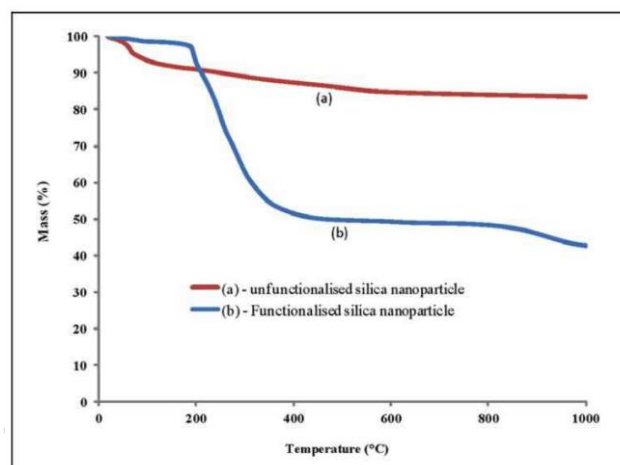


Fig. 10. thermal stability in terms of mass (%) content to temperature (%)

Figure 10. above shows that the thermal stability in terms of mass (96) content for functionalized silica (Figure (b)) has been reduced significantly compared to silica nanoparticles that were not functionalized (Figure (a)). This is because of the degradation of organic functional groups at lower temperatures (below 370°C) for functionalized silica. Thus, the TGA data confirm the attachment of functional groups on the silica surfaces. The effective silica concentration is lower with functionalized silica compared to the unfunctionalized ones due to the presence of functional groups from dual-step surface modification.

VI. CONCLUSIONS.

- The goal of the ongoing effort is to create a multifunctional hybrid configuration using different nanoparticles made of titanium, nickel, and silica-based ceramics. Since the majority of the procedure is solvent-based, a desired network of the selected modifier and previous metals was created.
- The most appropriate approach is the one that is now being employed, which is a modified Stober's method. As can be seen, the mechanical and physical qualities have been significantly improved.
- The bonding, crystal structure, and compositional variation of the derived particles exhibit mesoporous structure with nanosized particle range as well as near net uniform porosity throughout the network of the powder system with all the metals attached to the silica as bonding through hydroxyl and carbonyl group.
- Due to the ability of the material made from sol-gel to adjust the appropriate features for applications. These powders are cast to the new shape as needed in a specific component to make them suitable for aeronautical and automotive applications.
- Another intriguing finding is that the combination of nickel and titanium produced Ni-Ti, or nitinol, which has greater super elasticity at peak temperatures and allows desirable powders to maintain mechanical strength at high temperatures.
- This essentially happens at a certain composition, which is reported in the literature to be close to equiatomic, and these minute traces in the XRD and FTIR are implicative and can benefit aerospace applications.

REFERENCES

- [1] J. Zhou, X. Yuan, W. Xing, W.J. Si, S.P. Zhuo, 2010, Capacitive performance of mesoporous carbons derived from the citrates in ionic liquid, Carbon, 2765-2772.

- [2] W. Xing, C.C. Huang, S.P. Zhuo, X. Yuan, G.Q. Wang, D. Hulicova-Jurcakova, Z.F. Yan, G.Q. Lu, 2009, 1--ierarchical porous carbons with high performance for supercapacitor electrodes, Carbon 47, 1715—1722.
- [3] W. Xing, S.Z. Qiao, R.G. Ding, F. Li, G.Q. Lu, Z.F. Yan, H.M. Cheng, 2006, Superior electric double layer capacitors using ordered mesoporous carbons, Carbon 44, 216-224.
- [4] J.H. Balthis, P. Mendenhall, 1952 , Preparation of sols from finely divided silicon, USP 2614994.
- [5] Y. Kobayashi, J. Imai, D. Nagao, M. Konno, Colloids Surf. A 326 (2008) 109114.
- [6] H. Wang, M. Yu, C.K. Lin, J. Lin, J. Colloid Interface Sci. 300 (2006) 176-182.
- [7] R.R. Iler, The Chemistry of Silica, Wiley, New York, 1979.
- [8] H. Hassander, B. Johansson, B. Törnell, Colloids Surf. 40 (1989) 93—105.
- [9] K. Unger, J. Chromatogr. 359 (1986) 61.
- [10] T. Suratwala, R. Steele, M.D. Feit, L Wong, P. Miller, J. Menapace, P. Davis, J. Noncryst. Solids 354 (2008) 2023-2037.
- [11] S.K. Park, K.D. Kim, H.T. Kim, Colloids Surf. A 197 (2002) 7-17.
- [12] S. Sridhar, D.H. Rasmussen, F.P. Chen, R.K. Kannabiran, Colloids Surf. A 132, (1998) 45.

