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Process and Development of Nano-Bio Composite Materials for the Bone Research

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Abstract

In this research work, a novel approach has been applied with respect to powder metallurgy route for the development of aluminium–graphene nano–bio composite materials for the application in bone research. The applied technology has been executed to synthesize Aluminium-Graphene composite materials with the help of high energy ball milling process followed by vacuum sintering to get the required devices. The fabricated devices and synthesize composite materials at later stage have been evaluated in terms of different micro-structural characterization tools like FE-SEM (Field Emission Scanning Electron Microscope), EDAX (Energy dispersive X-ray analysis) etc. for qualitative and quantitative analysis, which shows well distribution of the constituents of Al-Grapheme composite materials over the scan areas. The physical property of the sintered samples has also been evaluated through density measurement and it is found to be ≈ 97.5% density after sintering. The thermal fusing of Al based materials at the optimum temperature of sintering is found to be satisfactory with required strength. These nano-bio-congruent composite materials, which is carbon based can instigate and assist stem cell improvement and differentiation into diverse lineages. Moreover, the 2-dimensional material, graphene has the potential propensity to stimulate and escalate osteogenic differentiation, which makes it an exciting material for the bone regeneration research.

Keywords: Bone research • EDAX • FE-SEM • Graphene • Powder metallurgy

Introduction

Graphene has been successfully considered and is implemented for the realms like paint, coating, energy, biomedical, electronics etc. Bone tissue regeneration is of primary attraction to encourage faster relieved and reconstruction of huge bone defects produced by skeletal irregularity, fractures, tumor resection, and infection. The growth of this discipline needs the utilization of suitable substrates that can allow escalation, cell attachment, and differentiation. There are different kind of materials that can commence, trigger, and succour the series of complex events that can lead to cell osteogenesis and differentiation. For instance, collagen can provide appropriate surface chemistry for cell growth and differentiation but owns not so good mechanical properties and is susceptible to immune reaction. Hydrogels with adaptive chemical and physical properties may consequently direct stem cell fate. However, their restriction may include lack of cell-particular and specific bio related activities and it is demanding to produce huge structures due to the requirement of a highly cross-linked network that can make possible implications in cell behaviour. Hence, materials with inherent properties and characteristics that can maintain cell growth and instigate differentiation possess a great prospective for stem cell research. The Graphene is a 2-dimenional material which can be considered as single atomic sheet of sp_2 bonded carbon atoms. It is the lightest and maybe the strongest ˜material knwn to the scientific community. The conductivity behaviour and charge carrier mobility exceed the most conductive polymers by many orders making graphene an interesting material for electronic devices. Due to the advantage of functionalization, graphene has great prospective in biomedical and bone research application, also find many useful implications in the field of biosensors, drug delivery etc. As graphene can be synthesize d in a relatively pure form and offers tunable surface, it has emerged as an interesting substrate for experiments with anchorage-dependent cells such as Mesenchymal Stem Cells (MSCs), Meuronal Stem Cells, induced pluripotent stem cells, and others. There are different focus areas to use aluminium based materials in applied technologies.

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Indeed, graphene is one atom thick carbon material is a new form of carbon. Graphene exhibits remarkable thermal and electronic properties compared to the conventional materials such as aluminum, copper. The attractive properties of graphene (Gr) include the atomic thickness of material structures which is not possible in traditional materials, long mean free path of carriers, high carrier mobility at room temperature, minimal scattering due to defects because of the dirac particle nature of carriers, highest thermal properties compared to any materials known so far and is stronger than steel. Also, graphene has magnificent properties in terms of high Young's modulus (up to 1 TPa), high value of fracture strength (~125 GPa) and enormous thermal conductivity (~5000 W/m/K). That is why, graphene has been considered as a suitable reinforcement material for the synthesis and development of metal based nano-composites [1,2].

The advantage of sheet like structure of this 2D material (graphene) with larger surface area make it also an ideal candidate for the improvement of overall properties for the metal matrix nano-composite [3,4]. If we consider the improvement of the mechanical properties of the graphene reinforced metal matix composite, then it all depends upon how well the reinforced materials are well dispersed in the metal powder. As per the literature [5] it has been described a high mechanical hardness of 2–3 GPa (enhanced by 97% of the pristine Copper) for Copper-Graphene electrodeposited composite foils. One research group [6] have developed a different methodology with the help powder metallurgy using flake and consolidated sintering in argon atmosphere.

This is complied with extrusion in the hot condition to prepare Aluminium-graphene nano-sheets composites and has been detailed with improvement of certain mechanical properties. Another group [7] has communicated the report regarding surface moderation using Powder Metallurgy (PM) with the help of flake. The route of flake powder metallurgy, however, comprises graphene-oxide nano-sheets as the constituent material because numerous epoxy and hydroxyl groups reside on the surface of graphene-oxide, which promotes easy way in solvents and configure better steady solutions than grapheme [8,9]. Recent research [10] has also reported synthesis of Copper-Nickel nano-particle-based reduced graphene nano-sheets using graphene-oxide as the precursor under the *in situ* low temperature condition. It is well analyzed that, with the progress of research, Metal Matrix Nano Composite (MMNC) has been emerged as one of the paramount materials in twenty-first century.

The MMNC has been better chosen taken into consideration of magnificent properties in comparison to metals with unreinforced entity and provides potential way materials having higher strength, lower coefficient of thermal expansion, better result in terms of creep test and higher resistance to thermal fatigue [11,12]. MMNCs have made significant contribution in the applied field of automobile industries and electronic packaging [13,14]. Recently, MMNCs system with reinforced nano-elements has propelled a great interest to numerous researchers [12,14,15].

Till now, not much research has been carried out for the preparation of aluminium-graphene nano-composite and on the considearation of graphene dispersion in the metal matix. As this system of composite materials devised of combination of two or more constituents, that are primarily insoluble with each other, the synthesis of the same by ball milling process makes ideal platform to get required result. In this research work, the Aluminium-Graphene composite materials have been prepared using high energy ball milling process followed by vacuum sintering to get the required devices for the bone research.

Materials and Methods

Synthesis of Al-Graphene nano-bio-composites

The raw materials like: aluminium powder, graphene powder etc have been procured for the experimentations. The approach has been based on theory, modelling, simulations and experiments. Using state of art methods based on physics model such as density functional methods, molecular dynamics, the experiments were designed. This has not only provided guidance to experiments but also helped in reducing the cost and time involved in experiments. Incorporation of graphene intended to be tried in multiple ways. Both solutions based method, mechanical fine brushing as well as spin coating and preparing composites. The pristine aluminium powder (99.9% purity) and research grade graphene of are 3-5 layered aggregates of the sub-micron sheet with a lateral dimension of 5-10 microns and less with high aspect ratio about 100, more than 99% carbon content along-with natural presence of other entities were purchased from commercial source. The analysis was carried out to investigate the morphology and presence of materials with respect to Field-Emission Scanning Electron Microscopy (FE-SEM), Energy dispersive X-ray spectroscopy (EDAX), Transmission Electron Microscopy (TEM), and Raman Spectroscopy etc. for the Al and Graphene.

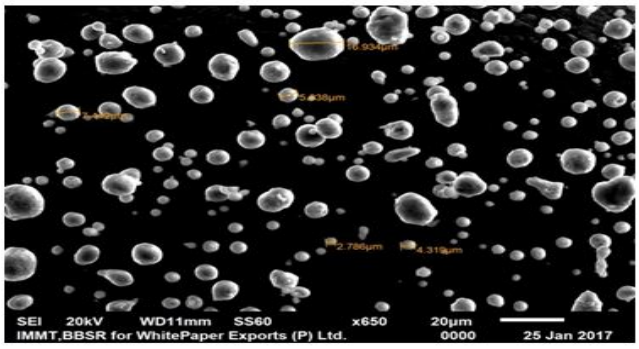

Figure 1. FE-SEM micrograph of Al powder.

Figure 1 shows the morphology of Al powder revealing the particle size in between 1 µm-5 µm with spherical size distribution in the given scan area. The constituent of the powder has been cross checked by EDAX, showing maximum% Al in the Figure 2.

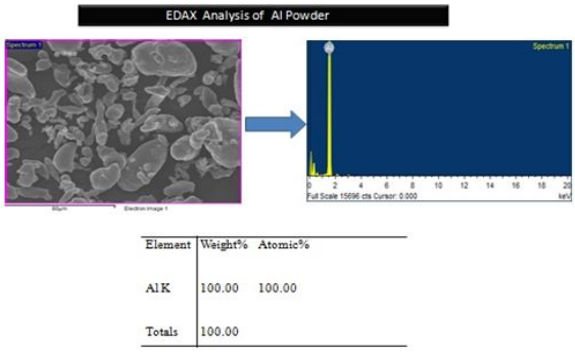

Figure 2. EDAX analysis of Al powder.

Both Figure 1 and Figure 2 confirm the qualitative and quantitative analysis of the Al powder. The intensity of the secondary electrons from the specimen working at low electron acceleration potential has a linear co-relation with respect to quantitative approximation of graphene layers. The required evaluation of the layer thicknesses is acquired with the help of secondary electrons discharged from the specimen with the help of low-energy electron detector. Transmission electron microscopy is also a very helpful characterization technique to examine the number of layers on checking the edges of the specimen, corresponding to a dark line with respect to each layer. The characterization is shown in the Figure 3.

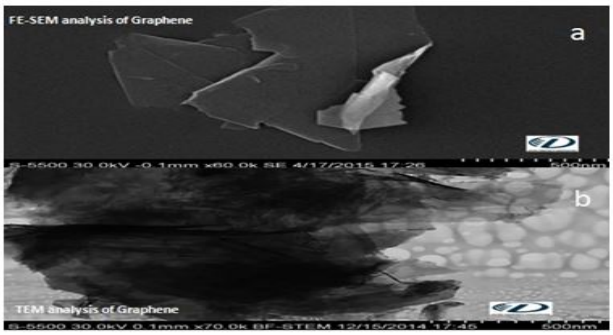

Figure 3. FE-SEM and TEM analysis of graphene.

The graphene has 3 primary major bands in raman spectrum. The location of D-band is closely to 1300 cm^{-1} which is considered as defect-induced band. The G-band location tends to 1580 cm^{-1} which is due to in-plane vibrations of the sp_2 bonded carbon atoms. The 2D-band is generally at 2700 cm^{-1} results from a second-order process. The existence of the D and 2D bands are in close relation to the double resonance raman scattering phenomenon and with increasing in the number of layers, the 2D-band gets broadened and blue shifted.

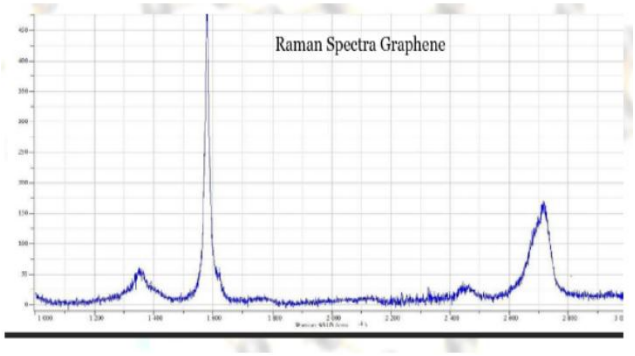

Figure 4. Raman analysis of graphene.

In our case, we have respective D, G, and 2D band in the graphene sample, as shown in the Figure 4. In this research work, the powder samples as per their respective weight ratios were taken in two different Stainless Steel (SS) jars along with Stainless Steel (SS) balls in the presence of toluene as a process control agent. The balls to powder ratio was maintained in 10:1. The milling was carried out for 10 hours with 300 rpm in high energy ball mill. After the milling, the Al based composite Powders were synthesized. The Sample details and the composition for the preparation of aluminium-graphene (Al-Gr) nano-composites are shown in the Table 1.

Mixing is an operation of blending of various powders of same creation or different grains of same powder while blending is limited to thorough blending of powders of more than one material; however both the terms are ordinarily utilized for any of the procedure. Al-Graphene powders were synthesized by this mechanical mixing process. This process of mechanical mixing manufactured uniform

Table 1. Sample details and composition for the preparation of Al-Gr composite.

SL. No.	Sample details	Composition (for 20 gm batch)	
		Aluminium powder (gm)	
1	Al	20	1
2	Al99.75Gr0.25	19.95	2
3	Al99.5Gr0.5	19.9	3
4	Al99.25Gr0.75	19.85	4
5	Al99Gr1	19.8	5

mixture of Al and Graphener. Al-Graphene powder as per the batch synthesis (in this case we use 20 gm batch) were placed in the ball milling set up consist of stainless steel balls 10 mm diameter, the process of mixing is continued for a duration of 10 hr at 300rpm in order to get uniform mixing for different composition of Al/Graphene materials or composites. The experimental set up for high energy ball bill is depicted in Figure 5a. Figure 5b shows the Stainless Steel (SS) balls of 10 mm diameter, Figure 5c is the stainless-steel jar with the cover, Figure 5d shows SS balls with SS jar and experimental instrument (make: in smart) for carrying out ball milling experiment, shown in the Figure 5. Before the experiment, necessary care is taken to clean all the SS balls along with SS jar with de-ionized water and acetone, so that no residues and foreign materials will be present during milling operation. This methodology greatly influences the uniformity and distribution of elemental constituents, as result of which Al powder and graphene powder make required composite materials after milled for 10 hours.

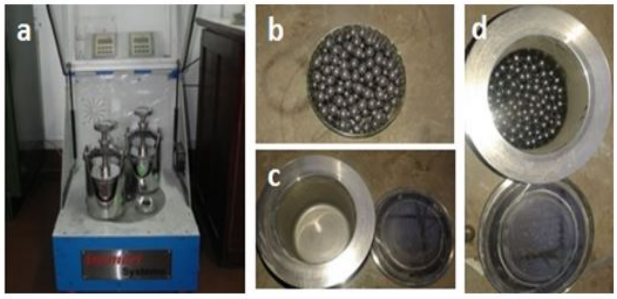

Figure 5. High Energy Ball-mill experimental set up.

The green pellets have been prepared using Al-graphene composite materials with the help of 10 mm cylindrical die using Hydraulic press machine. During the compaction 1.28 ton load was applied with holding time of 2 minutes. The green pellets then subjected to vacuum sintering at 630^0C for 2 hours with heating rate of 5^0C/min. The thermal fusing of Al-Graphene nano bio-composite materials at this temperature was found to be satisfactory with required strength. The Sintered Al based devices were finally fabricated. In this methodology, compaction is applied as it is a procedure by which the metal powders are squeezed into compacts of craved shape with satisfactory quality to withstand discharge from the devices and ensuing taking care of culmination of sintering with no breakage. The work part after pressing is called compacted green specimens. Sintering below the melting point of aluminium is considered as dynamic move occurring without liquefying from the condition of metallic particles to enormous state which ought to be free from porosity and have intact physical and mechanical properties. Sintering resulted in solid holding between particles bringing about quality, densification and dimensional control beside grain development, arrangement of grain limit and shutting of voids present in the grain. Even mechanical quality, pliability, electrical and warm conductivities are favoured by expanded measure of sintering. The compacted specimens were placed in the vacuum sintering furnace with heating rate of 5^0C/min for 2 hours at a temperature of 630 degree Celsius, as the melting point of Al is about 660 degree Celsius. After the sintering process over, the sample chamber was cooled for 12 hours and put in the vacuum condition after which the sample is taken out for characterization.

The Schematic diagram for illustrating synthesis of Al-graphene nano-composite and fabrication of sintered devices are depicted in Figures 6a and 6b respectively.

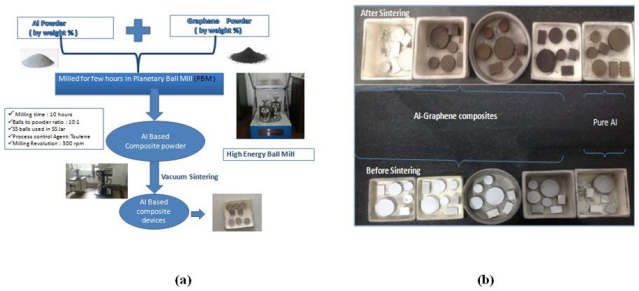

Figure 6. (a) Schematic diagram of synthesis of Al-Graphene composite materials and device fabrication (b) Al-graphene sintered devices.

Results and Discussion

Characterizations

The composite materials Al99.5Gr.5 and Al99Gr1 have been subjected to morphological analysis with the help of Zeiss, Carl Zeiss SMT AG Field Emission Scanning Electron Microscopy (FE-SEM) attached with Energy Dispersive X-ray spectrometer (EDX) for the micro-structural characterization of the prepared composite materials and sintered pellets.

The FE-SEM is a kind of electron microscope, where the electron energy is utilized for morphological analysis of the samples by scanning it with a beam of electrons. The electrons interact with the specimen generating signals that contain data about the specimen's surface morphology, composition etc. In our case, the characterization was carried out using the interaction of secondary electrons with the samples. As shown in the Figure 7, both the composite materials Al99.5Gr.5 and Al99Gr1 exhibit spherical grain size distribution with individual particle sizes <10-20 nm. The distribution shows the uniformity of Al and graphene constituents and is concluded to be nano-composites with spherical in shape, uniform in size and homogeneous in doping which are the requirements for its use as an electrode for the application in battery, solar light devices etc. The prepared composite materials have been found to be satisfactory as can be seen from Figure 7. The quantitative analysis of these composite materials through elemental mapping and EDAX are shown in the Figure 8.

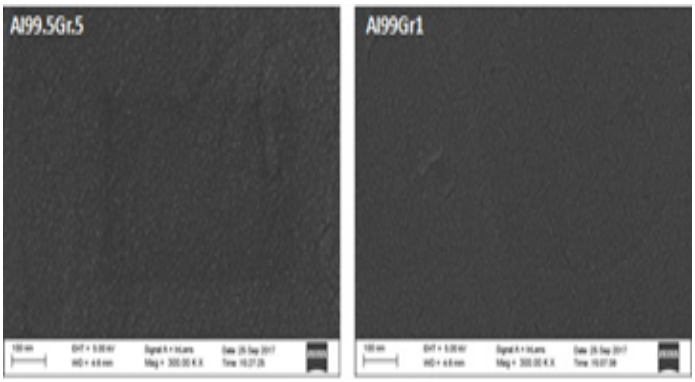

Figure 7. FE-SEM analysis of Al99.5Gr.5 and Al99Gr1 composite materials.

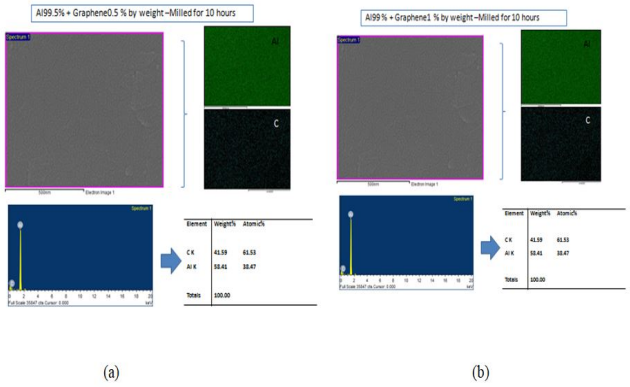

Figure 8. Quantitative analysis of Al99.5Gr.5 and Al99Gr1 composite materials.

of 10% to 102% I.A.C.S (International Annealed Copper Standard) with an accuracy of+/- 1% IACS. The probe impels eddy currents at a constant frequency in the test part. These current influence the electrical impedance of the test probe. The variation in impedance is proportional to the electrical conductivity of the test part. Thus, conductivity measurement is possible by measuring the corresponding change in probe impedance. Before the use, the instrument is set against standard "High" and "Low" conductivity sample provided with the instrument. The probe is placed on a flat surface of the test part. The change in the probe impedance is sensed and processed and the conductivity of the test sample is expressed as % IACS on digital display. The Instrument is compensated against "lift off" effect. Thus, the errors produced, due to roughness of test surface up to 0.05 mm are eliminated. The off-set compensation switch helps in compensating drift due to temperature effect.

Table 2. Density measurement before and after sintering.

SL. No.	Sample details	Composition (for 20 gm batch)			
		Alumnium powder (gm)	Graphene Powder (gm)	Density (%) before sintering	Density (%) after sintering
1	Al	20	0	95.18	98.88
2	Al99.75Gr0.25	19.95	0.05	84.37	97.56
3	Al99.5Gr0.5	19.9	0.1	85.02	96.88
4	Al99.25Gr0.75	19.85	0.15	86.45	97.19
5	Al99Gr1	19.8	0.2	87.62	97.74

Measurements

Density analysis: The physical property of the sintered samples (10 mm diameter and 3 mm thickness) was examined *via* density measurement. Using Archimedes principle, the bulk density of the sintered samples was evaluated by measuring the weight of the sample in air, weight of the sample in water and taken into consideration of density of water and it was then finally compared with the theoretical density. The theoretical density was calculated using simple rule mixture. In our methodology, we checked 4 sintered samples for the purpose density measurement before and after sintering. This time, after sintering we find ≈ 97.5 % density for all the samples, which implies a very good indication for the continuity of the sintered samples for the conductivity measurements. Table 2 shows the density of the samples before and after sintering.

Measauarement of electrical conductivity: The electrical conductivity of the sintered samples had been examined through Conductivity Meter –Type 979, which is based on the principles of eddy currents. It measures electrical conductivity of non-ferrous metals in the range

The probe is 12 mm in diameter and can be used in any reasonable flat accessible surfaces. In our case, our sintered pellets are 10 mm in diameter and 3 mm thickness; hence the probe can have easy accessible and have good contact surface area over our samples. With respect to the Al and Al-Graphene nano bio-composite devices, which have been prepared through powder metallurgy route, the electrical conductivities of the samples have been investigated at room temperature, 100°C and 200°C through eddy current methods as per the sample dimension requirements of the measurements. The findings are as follows. It has been found that at higher temperatures, the electrical conductivity follows the same trend like at room temperature i.e, with increase in weight % of graphene into the Al system, the electrical conductivity value is increasing as shown in the Figure 9. However, it has been further interpreted that, the room temperature electrical conductivity is more than at high temperatures at 100°C and 200°C. The repeatability has been checked and the results are quite reproducible every time. Hence, the electrical conductivity of these nano bio-composite materials can be well applied in bone tissue

engineering to expedite cell growth and tissue rejuvenate with physioelectrical signal transfer.

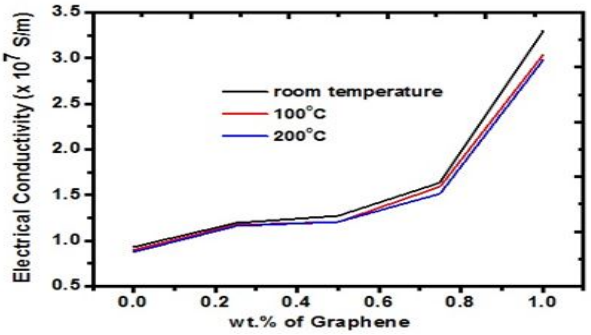

Figure 9. Electrical Conductivity of Aluminium-graphene nano bio-composite.
Note: (—) room temperature, (—) 100 ^{0}c, (—) 200^{0}c

Conclusion

In this research work Aluminium–Graphene nano-bio composite materials have been synthesized for the bone research using powder metallurgy route and the devices of the same were fabricated through vacuum sintering and the analysis with respect to morphological and compositional analysis were done. The composite materials have shown spherical grain size distribution with individual particle sizes <10-20 nm. The distribution shows the uniformity of Al and graphene constituents and is concluded to be nano-composites. Also, the composite materials have been found to be uniform distributions with respect to Al and C (graphene) over the given scan areas and the weight % and atomic % of Al are more than C. These composite bio materials can be used for bone repair or regeneration due to its chemical analogous to that of natural apatite in bones. The electrically conductive these nano biomaterials can be well applied in bone tissue engineering to ease cell growth and tissue rejuvenation with physio-electrical signal conduction.

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