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PLA Based HA Added Build Material for Rapid Prototyping Technologies

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Abstract: Rapid prototyping is a huddle of techniques used to create scale model of a part or assembly using CAD model. The nature of RP technique allows the user to create complex and complicated profiles in the form of prototypes. There is a possibility to use RP systems to create end products, for which the selection and properties of the work material plays a major role. This paper investigates the possibility of using Polymer based work material for Rapid prototyping. Polylactic acid [PLA] is a bio polymer which has been already used as a RP work material, in which Hydroxyapatite [HA] is added along with Aluminum oxide [Al_20_3]. This paper discusses the synthesis of HA and prepares the specimen by mixing PLA along with Al_20_3 adaptable to RP Techniques. The suggestive materials mechanical characteristics are also presented.

Key words: Rapid Prototyping · Polylactic acid · Hydroxyapatite

INTRODUCTION

Rapid Prototyping (RP) is a technology which works on the principle of additive layer manufacturing. ASTM Standard defines Additive manufacturing (AM) as 'the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies, such as traditional machining'. The nature of additive layer manufacturing technique enables the system to make complicated profiles and intricate shapes which are not usually possible in traditional manufacturing processes. The CAD model of the desired product is given as an input. Then the CAD file is sliced into small layers which are referred as Standard tessellation file (.stl file format). In general, the '.stl' file determines the orientation of the tool head in a RP system. There are many techniques employed this process. Some of the popular processes are Stereo lithography (SLA), Selective laser sintering (SLA), Fusion deposition modeling (FDM), Ballistic particle method, 3D Printer (3DP) etc. Even many techniques available in AM, each techniques having their own merits and demerits towards the manufacturability of the product [1].

In most of the cases, Additive manufacturing is extensively used to make prototypes that are used to check the ease of assembly (fit function) or functionality checking of the product. The prototypes created by RPT used as an aid to communicate and inspect the tools, so that any deviation can be addressed in the earlier stage hence time and cost will be saved. Also the working methodology of RPT shows that it will reduce the production development sequences [2].

Since RP techniques stands good for produce complex shapes, intricate profiles it has a confined restriction towards the work material. Many of the RPT are not accessible to work with common engineering materials which yield satisfactory mechanical properties (Ex: Metals, Polymers, Composites & ceramics) [3]. It induces interest among researchers to explore themselves to investigate new materials for RP systems. Also the process parameters like Temperature, orientation, feed rate are also plays a major role along with selection of material. RM opens a wide spectrum in manufacturing field by making products which are usually not possible in other systems with ease of manufacturability [4].

The steps followed in a standard AM are given below:

- Conceptualization and make CAD model
- Conversion of cad file into a.stl
- Transfer and manipulation of.stl file on AM machine
- Setup the AM system
- Build the first layer
- Build subsequent layers
- Part removal and cleanup
- In case of requirement go for Post-processing
- Application

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In the subsequent section, basic theories related to various techniques are emphasized in a brief manner.

RP Techniques – An Overview: RP systems works based on the working principle of Additive layer manufacturing, in particular bottom-up approach. It finds application in Medical devices, Die set, Aerospace and also facilitate customer to make customized products.

RM widely used to make consumer products, also find application in making electronic items [4]. The aerospace sector has also found a number of applications, often driven by the possibility of improving buy-to-fly ratios (as some AM processes have high material utilization and reducing the weight of components through design optimization[5]. RP systems find applications in engineering and analysis sector: Validation of CAD model, to envision the object, to evidence the concept, presenting and promoting the model, fit and form models, flow characteristics study, distribution of stress, spot and surgical operation planning, design and fabrication of customized prosthesis and implanting are the fields where. In manufacturing sector it is used to support Vacuum casting, Metal spraying, Investment casting, Die casting and used to make EDM electrodes, Master models etc. for the industrial arena.

As mentioned in the earlier section Stereolithography (SLA) is a technique uses a resin which should be photo curable, i.e. it should solidified when it is exposed to a controlled environment. A specified wave length light has to be focused on to the resin surface as per the desired orientation, in which the larger molecules obtained because of polymerization of smaller molecules. The subsequent layer can be achieved by the appropriate table movement in order to get the prototype. Among AM technologies, Selective Laser Melting (SLM) has become a relevant production method for manufacturing ready-to-use parts made from metals such as stainless steel, nickel, titanium and aluminum alloys.

Another technique called Laminated object manufacturing (LOM) used where the laminated sheets are used as a work material. A pre-glued sheet spread over on a table in which a laser beam is cuts the contour of the desired profile. Several such profiles when glued or welded will yield the prototype.

Selective laser sintering (SLS) is another technique which is similar to SLA, but the work material is a powder. A laser source traces the desired cross section of the powder surface and the surface is fused and solidified. Subsequent layer of powder is spread over on the top of the solidified. Again the laser traces the desired cross section and the process continues until get the required component.

In Ballistic Particle Method, an inkjet mechanism is used to discharge droplets of molten materials which get cold-welded together on a earlier deposited layer. A layer is created by moving the droplet nozzle in X and Y directions. The base-plate i s lowered b y a specified distance after a layer is formed and a new layer is created on the top of the previous one; finally the model is created.

Even RM is used widely in manufacturing arena to make high end functional products with high accuracy and specifically it finds application in making a prosthetics and implants. But the products like tool inserts, moulds and dies which are usually made with high strength material find restricted application to use RM [6]

Still RM opens its gate to the world of Polymers and ceramics to some extent, feasibility for metallic components shows poor result. One of the main reasons for that is to attain fine raw metallic materials (Approximately the particle size should be equal to 20 - 100 μm); and to bind the powders layer by layer to get solid products is difficult. And getting the product with desirable electrical and thermal properties are still difficult. Achieving higher accuracy and surface finish is also still a challenging task [6]

It will open a new atrium when the RM systems allow the very high strength materials as its work material. It will allow making high end surface finish and accurate elctro mechanical components, implants and prosthetics [6].

The desired porosity levels can be achieved by controlling the process parameters such as flow rate of the raw material i.e. powder and size of the particles [6]

RPT Material: The output of RPM technology is mainly depends on the kind of material which is used to make the product. Even the material satisfies the working range for a RP system it should also ensure the sufficient mechanical characteristics and few other functions after forming [9].

One of the major problems addressed during the process is that it will absorb the moisture through the polymer feedstock. It results in to serious problems when extruding the parts. Because of that the morphology of the material getting affected and also it will obstruct the print nozzle. Also bubble formation and bulges caused over on the surface of the product [10].

In few cases the temperature exceeds a certain limit where it will results in degrading the polymer, the breakage of polymer chain, yield poor strength product and left with residue in the melt sections [11].

In SLS, the particles are joined together by the action of melting and solidification. It is actually binding similar to “Gluing” by an organic binder with an appropriate controlled temperature. Usually IT will be lesser than the melting point of the material. temperatures during SLS [12].

These ever-increasing demands of the MMCs are mainly due to their superior mechanical properties such as higher transverse strength and stiffness, greater shear and compressive strength and better high temperature capabilities [13].

But for FDM processes, thermoplastics finds a wide application as a work material.A plastic which will be softened under a given temperature if refereed as thermo – softening plastics are used in this process. Because of the applied heat it squeezed and become liquid and sintered after freezing and cured to attain a solid state [14].

But more stress will be accumulated in the created part because of uneven heating condition and also it results in distortion which primarily affects the strength of the product. It usually ends up with poor strength and shorter life time of the product [14].

In this work, FDM process has been chosen to investigate an alternative build material, expected to overcome the above mentioned drawbacks.

The basic working set up and operational sequence of a FDM is explained in this section. In this technique, a spool of thermoplastic filament is fed into a heated FDM extrusion head. X and Y movements are controlled by a computer so that the exact outline of each section of the prototype is obtained. Each layer is bonded to the earlier by heating. This method is ideal for producing hollow objects.

In this technique, the object is made by squeezing a continuous thread of polymer through a narrow, heated nozzle that is moved over the base plate. The thread melts as it passes through the nozzle, only to get hardened again immediately as it touches (and sticks to) the layer below. A support structure is needed for certain shapes and this is provided by a second nozzle squeezing out a similar thread, usually of a different color in order to make it easier to distinguish them. At the end of the build process, the support structure is broken away and discarded, freeing the object. The FDM method produces models that are physically robust. Wax can be used as the material, but generally models are made of ABS plastic.

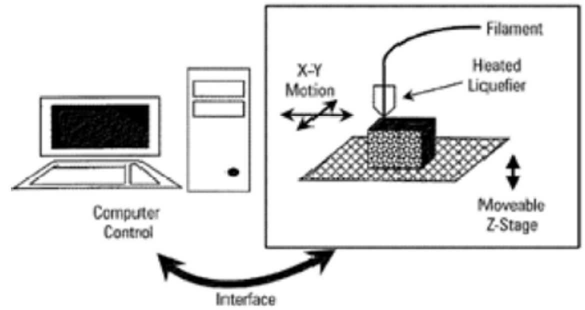

Fig. 1: Schematic diagram of FDM

The nature of this process excites the researchers and academicians all over the world and they show interest to improve the system performance in the entire possible manner. [15].

Polymers such as Elastomers, Thermoplastics and waxes find a wide application in FDM process. As same as the other RM techniques the products created by this process are also used to check the functionality, ease of assembly and to and verify the size and shape. Since the FDM process narrow down its application towards the build materials, this work focuses to investigate and study its characteristics for this process. And it is believed to have an increase in usage of FDM to enhance RM systems. [16].

The FDM system allows creating new materials including composites in order to facilitate good properties and required strength [17].

To develop an alternate material for FDM fascinating researchers and many progresses has been shown but in a slower manner. Developing a new material for FDM to give enough properties requires a profound knowledge in material science arena. Many initiatives started to use ceramics and metals as a build material and come with some satisfactory results.

Application of ceramics and metals for FDM is carried by Rutgers University in the United States. They have has put work in the development of fused deposition of ceramics (FDC) and metals. Silicon nitrate, Aluminum oxide, hydroxypatite and stainless steel are used in their work for to fabricate different types of structural and bio ceramic applications. They create such components on the FDM using ceramic powders mixed with organic binder system. The properties of the mixed feedstock ?lament meet the ?exibility, sti?ness and viscosity required for successful FDM processing. But the fabricated green parts need to undergo further processing to remove the organic binder and are subjected to sintering to achieve densi?cation. Sintered part may be infiltrated with other type of metallic materials [18].

Material properties such as strength, electrical and thermal conductivity and optical transparency typically have inferior properties compared to conventionally manufactured counterparts due to the anisotropy caused by the layer-by-layer approach. Therefore, application options within these materials genres are usually limited to models for form/fit testing, functional testing, presentation models, prototypes and non-load bearing products. Overcoming these issues will require advances in both process control approaches and material selection [19].The field of RP is still developing, with much effort being expended on improving the speed, accuracy and reliability of RP systems and widening the range of materials for prototype construction. However, in future the focus may be to produce parts as end-use products. In this context the term 'rapid manufacturing' (RM) is used in which RP technologies are employed as processes for the production of end-use products. The introduction of non-polymeric materials, including metals, ceramics and composites, represents a much anticipated development [20].

Researchers innovate and validate new materials including composites and find that the tensile modulus and strength of this material were approximately four times those of ABS. Therefore, prototypes fabricated with these materials would have greater functionality than those fabricated with ABS[21].

The FDM technology thus offers the potential to produce the functional parts with a variety of materials including composite materials. But little work seems to have been done in the development of metal/polymer composites for direct rapid tooling application using the FDM system. Direct rapid tooling of injection molding dies and inserts can be conveniently performed if strong metal based feedstock material is available for the FDM rapid prototyping systems.

This paper presents the development of a new material based on sol gel technique for FDM process. The research focuses on developing proper formulation and mixture of constituent materials for obtaining to satisfy certain properties hence they can be produced in filament form for use in the FDM process. The success of this unique work depends upon careful selection of proportion of constituent materials and fillers to result in the desired properties of the composite material. The main outcome of this major breakthrough is the manufacture of strong, flexible and spoolable feed stock filament made by extrusion from this composite material for use in the existing FDM system without any hardware or software modification.

Hydroxyapatite and Synthesis – An Overview: Hydroxyapatite is a naturally occurring mineral present in human bone in the form an of apatite group. Around 50% by volume and 70% by weight is a modified form of hydroxyapatite which is commonly known as bone mineral. The most important property with the hydroxyapatite is that it is stable on comparison with the calcium phosphate, besides hydroxyapatite is the most stable calcium phosphate compound under certain conditions when on calculating with temperature, pH and composition of the body fluids. The property that takes over the bone is that the chemical composition, chemical arrangements and alignment of the molecules. The hydroxyapatite is a porous composite that provides a support to the bone structure and gives a firm strength to the bones.

Since it is clearly inferred that the hydroxyapatite is a major contributed chemical compound that is present in bone in a large quantity that supports in giving a providing a support to the bone.

The most interesting factor is that the resemblance of the hydroxyapatite takes over the resemblance of bone as shown in fig.2, which means that the visual inference will be as similar.

The present goal of a natural appearance is finally achieved with the help of a natural material because of the remarkable similarities are noticed between the porous structure of hydroxyapatite and human bone.

This similarity has brought a mind-set that the alternative to the implanted titanium plate can be a hydroxyapatite composite. It is preferred because it shows a great medical supportiveness to the replacement bone structures.

The natural derivative material has the porous structure and the chemical structure of bone. Thus, the system of the body will accept to the natural composite implanted hydroxyapatite. This hydroxyapatite in combination with the certain chemicals will give a great advantage in giving a support to the bone implants.

At present HA is a solution for a number of medical applications regarding bone implants and bone draft. To list down all of its applications, its important material properties such as biocompatibility, its bioactivity and its mechanical strength should be determined and personalized. There are many researches that have been done and are ongoing regarding the development of the preparation methods that enables us to control the methods and thus get a controlled and desired chemical and physical characteristics of the powder.

Out of the many phosphate compounds that are available, the calcium phosphate system is the most highly detailed and also very complex. Most important fact that is usually overseen is that the purity and features of the particles of the final synthesized powder has an influence on the following properties and can change these: Bioactivity, biological dissolution and its corresponding properties. These properties determine the medical application of the material, thus also determining the type of synthesis method that is to be followed to prepare the compound. The control of properties such as crystal morphology, chemical composition, crystal structure, particle size distribution and agglomeration. Since many methods are available to synthesis HA, Wet chemical precipitation method has been chosen for this work.

Maintaining conventional wet chemical precipitation methods in the literature is one of the most extensive methods because of its use of relatively inexpensive raw materials. Other good factors also include its immediate availability and the simplicity of the material. Its low reaction temperature is also a very good property which results in very minimal operating costs. In addition, due to its scalability, this process is attractive to manufacturing applications. However, precipitation of HA cannot be considered insignificant due to nucleation, crystal growth, thickening and / or agglomeration at the same time. The reaction needs to be fine-tuned to optimize the morphology and minimize the growth of the crystal as shown in fig.3&4. In addition, various surfactants or dispersants have been studied to reduce particle aggregation because of high particle energy. A high temperature heat treatment in addition to the high particle energy is also required so that the percentage of crystalline phase is maximized during the precipitation process of calcium phosphate. This is very time consuming. To avoid this saturation is done. Super saturation is when the solution contains more solute than the solute that the equilibrium level as a result of which nucleation and crystal growth occurs. This occurrence occurs when the phosphoric acid solution is titrated in a calcium solution to form a suspension of precipitated particles. The final composition depends on the properties such as pH, concentration and temperature of the solution; therefore, these reaction parameters must be controlled to produce a homogeneous product. The precipitated powder that is formed is usually calcined. This is done to improve the crystal structure.

Fig. 2: Synthesized Hydroxyapatite

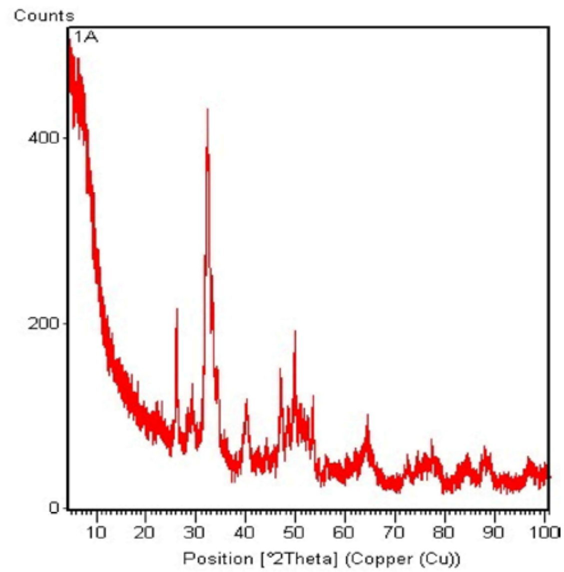

Fig. 3: XRD result for synthesized Hydroxyapatite

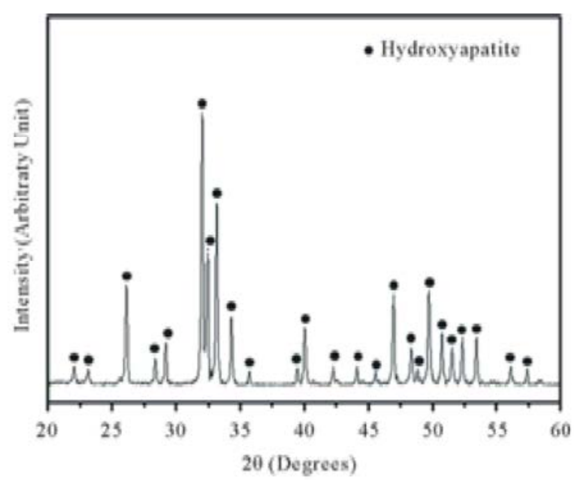

Fig. 4: XRD result for Hydroxyapatite

Mechanical Characterization of the Composite Material: Tensile testing is test in which a material is subjected to a controlled force of tension until the material breaks.

The parameters or properties that are directly determined in tensile test are: Tensile force, maximum elongation and ultimate tensile strength. With the help of these properties secondary parameters like Young's modulus, Poisson's ratio, yield strength and other strain-hardening characteristics can also be determined. In uniaxial tensile testing the component is stretched in any one direction. To obtain the mechanical properties of isotropic materials, the most commonly used method is uniaxial tensile testing.

In biaxial tensile testing the component is stretched in two different directions. For anisotropic materials, such as composite materials and textiles, most commonly used method is biaxial testing.

The specimen that is prepared for tensile testing is of a standardized cross-section. The specimen is of 2 parts: Two shoulders and the section in between the two shoulders. This in between section is called as the 'gage'. The gauge section of the specimen has a smaller cross section when compared to that of the shoulder. The failure occurs in this part of the specimen. The sizes of the shoulders at the two ends are larger than the cross section of the gauge part so that they can easily be gripped. The dimensions and the tolerances values of the test specimen should be followed as per standard ASTM E8.

The machine on which the tensile test is to be done should have the proper requirements for the test specimen that is going to be tested. There are four important parameters that of the testing machine: Accuracy, Speed, Precision and Force capacity.

Force capacity is the property of the machine which generates the force required to perform the tensile test. It should be capable of generating enough force such that the specimen is fractured and thus reaching failure fracture. It should also be capable of applying the force at different paces. It should be fast or slow enough depending on the specimen and its application as shown in fig.5.

A machine that is not accurate and precise will yield wrong results. Thus accuracy and precision are very important properties of testing machine.

The test specimen is placed in the testing machine and force is applied thus slowly extending it until it fractures. During this test, the elongation of the gauge section and the applied force is recorded and tabulated. Load vs. Displacement and Stress-Strain curves are plotted and the data is analyzed.

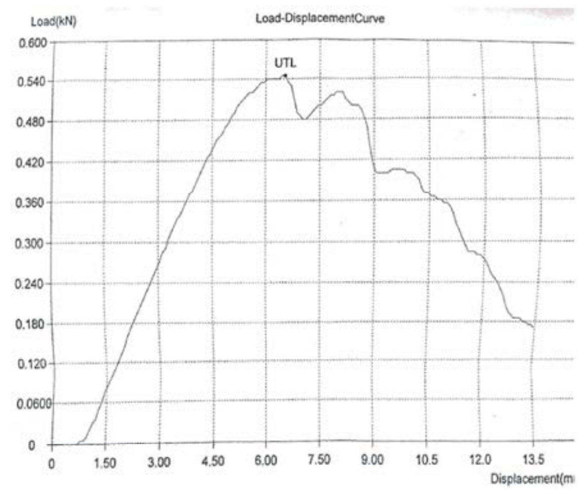

Fig. 5: Tensile strength result for the specimen

A compression test is a test where the test specimen is subjected to opposing forces that push the specimen inward upon the action of force from opposite sides. It can also be simply defined as the act of squashing, crushing, or flattening the material.

In compression the behavior or response of a material is recorded when it is subjected to a compressive load. Parameters such as strain, stress and deformation are determined from this test. The ultimate parameter that is determined is the compressive strength of the specimen. Also the compressive strength, yield strength, ultimate strength, elastic limit and the elastic modulus are some of the other properties that are determined. By determining these parameters and the values associated with it, the specimen may be determined whether or not it is suited for specific applications or if it will fail under the specified stresses.

The test specimen is placed in between two plates which distributes the load that is applied across the entire surface area of the two opposite faces of the specimen. After that the plates are pushed together by a universal test machine making the sample flat. The compressed specimen is shortened in the direction in which the force is applied and it expands in the perpendicular direction of the force applied. The compression test is more or less the opposite of a common tensile test.

In impact test an object's ability to resist high-rate loading is determined. It is a test where two objects strike each other at high relative speeds. The ability to resist impact of a specimen is one of the determining factors in the service life of the material. The suitability of a designated material for a particular application can also be determined. Impact resistance is one of the most difficult properties to measure. The ability to quantify this property is a great advantage in product liability and safety.

The impact strength of a material is its ability to absorb shock and impact energy without breaking. The impact strength is defined as the ratio of impact absorption to that of the test specimen cross-section. Toughness of the material depends upon the shape of the test specimen and its temperature.

In general Charpy and Izod tests are conducted to know the impact strength of the specimen. In Charpy test, the test specimen is supported at both the ends while in the Izod test, the test specimen is clamped only on one side.

The results of an impact test will infer Failure strength, breakage area strength and energy loss due to the impact.

Table 1: Impact strength result for the prepared specimen

Test Temp.	Notch Type	Sample ID	Specimen Size(mm)	Absorbed Energy-Joules
24◎	Un Notched	1	3.5×10×80	9.33
		2	5×13×80	14.67

CONCLUSION

This paper attempts to suggest that the possibility of polymer based HA as a work material for the additive layer manufacturing. Out of many RP techniques, Fusion Deposition Modeling technique has been chosen, because of the flexibility in the process and the adoptability lies between the suggested material and the process. Synthesis of HA takes in account, which constitutes a major role in the properties of the material. If the suggested material shows its feasibility related to the process, then it will explore a wide range of new possibilities in the world of product design and manufacturing.

Abbreviations:
3DP – 3D Printer
AM – Additive Manufacturing
RP – Rapid Prototyping
RM – Rapid Manufacturing
STL – Standard tessellation file
FDM – Fusion Deposition Modeling
LOM – Laminated Object Manufacturing
SLS – Selective Laser Sintering
SCD – Super Critical Drying
SLA- Stereo lithography

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