International Conference on Systems, Science, Control, Communication, Engineering and Technology 2015 [ICSSCCET 2015]

ISBN	978-81-929866-1-6
Website	icssccet.org
Received	10 - July - 2015
Article ID	ICSSCCET020

VOL	01
eMail	icssccet@asdf.res.in
Accepted	31- July - 2015
eAID	ICSSCCET.2015.020

Processing Techniques of Functionally Graded Materials – A Review

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Abstract- *Functionally Graded Materials (FGMs) are a class of engineered materials characterized by a spatial variation of composition and microstructure aiming at controlling corresponding functional (i.e. mechanical, thermal, electrical, etc.) properties. The tailored gradual variation of microstructural features may be obtained through non-uniform distributions of the reinforcement phase(s) with different properties, sizes and shapes, as well as by interchanging the role of reinforcement and matrix materials in a continuous manner. Wide ranges of processing methods are considered on the production of FGMs. Each processing method has its own characteristics on the gradation phenomena which implies on the product. Processing parameters and their influences are presented with consideration of experimental investigations carried out earlier in this field. Also the processing steps to attain the desired gradation with limitations are discussed. Microstructural evaluation, wear mechanisms, porosity, stress distributions, etc. of various metal-metal, metal-ceramic and ceramic-ceramic FGMs are discussed to expose an overall view for carrying future research. Finally the applications of FGMs in various fields, which are still facing new innovations are considered. Improving the performance of processing techniques and extensive studies on material characterization on components produced will go a long way in bringing down the manufacturing cost of FGM and increase productivity in this regard.*

I. INTRODUCTION

FGMs exhibit gradual transition in the microstructure and/or the composition in a definite direction, the presence of which leads to variation in functional performance within the part through microstructural manipulation. FGMs possess a characteristic of tailoring of graded composition and micro structure according to the distribution of properties needed to achieve desired function which distinguishes it from the conventional materials. As we all know composite materials and cermets have been employed as a solution for the various engineering problems for the number of years. Though the development of new materials (FGMs) is due to mismatch occurs while applying them as a coating on the surface of the base material to withstand desired condition which leads to change in the properties like elastic modulli, thermal expansion and hardness. The gradual transition allows the creation of superior and multiple properties without any weak interface.

According to the material composition function specified, the volume fraction of one material constituent will be changed from 100% on one side to zero on another side, and that of another constituent will be changed the other way around as shown in Fig.1. The FGM helps to reduce stress, prevent peeling of the coated layer, prevent microcrack propagation, etc. For a component having a material region made of an FGM, its fabrication technology must be able to add different materials with certain volume fractions simultaneously for every pixel according to the specified composition function.

Cite this article as: Saiyathibrahim.A, Mohamed Nazirudeen.S.S, Dhanapal.P. "Processing Techniques of Functionally Graded Materials – A Review." *International Conference on Systems, Science, Control, Communication, Engineering and Technology (2015)*: 98-105. Print.

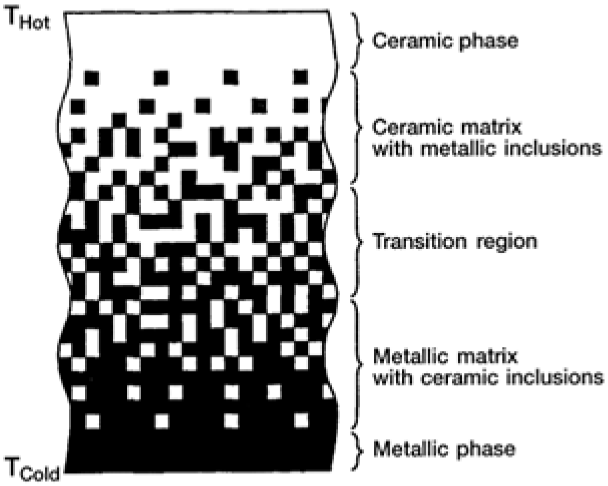

Figure 1. Structural view of continuously graded FGM

Various fabrication methods are available for the preparation of bulk FGMs and graded thin films. The processing methods are commonly classified into four ways like powder technology methods (dry powder processing, slip vesting, tape casting, infiltration process or electrochemical gradation, powder injection molding and self propagating high temperature synthesis, etc.), deposition methods (chemical vapour deposition, physical vapour deposition, electrophoretic deposition, slurry deposition, pulsed laser deposition, plasma spraying, etc.), in-situ processing methods(laser cladding, spray forming, sedimentation and solidification, centrifugal casting, etc.) and rapid prototyping processes (multiphase jet solidification, 3D-printing, laser printing, laser sintering, etc.)[1].

The manufacturing process of a FGM can usually be divided into two steps. Initial one is building up of the spatially inhomogeneous structure called Gradation. Another is the transformation of this structure into a bulk material called Consolidation. In detail gradation process can be categorized into constitutive, homogenizing and segregating processes. Stepwise build up of the graded structure from precursor materials is constitutive process. Homogenizing is a process of converting sharp interfaces between two materials into a gradient by material support. Segregation starts with a macroscopically homogeneous material, which is converted into graded material by material transport caused by an external field (i.e. gravitational, electrical field, etc). Normally sintering and solidification follows gradation process. Graded structure is of two types namely continuous and step wise. In continuous type structure, change in composition and/or microstructure occurs continuously with position. Powder metallurgical processed FGMs follow discrete (or) step wise structure. In detail their microstructure feature changes in a step wise manner with interfaces existing between discrete layers. Continuous graded structures are to be produced by centrifugal casting [2].

In this paper processing techniques of FGMs are presented with their experimental investigation of various materials. Some research works on functionally graded materials in recent times are presented and the future research needs are proposed. This work clearly demonstrates various possibilities available in FGM research and useful to gain background knowledge. Also some applications of functionally graded materials are presented here.

II. PROCESSING TECHNIQUES OF FGM

In this chapter a summary of the different technologies and process for the production of FGM is provided. This includes promising processes such as dry powder processing, Slip casting, tape casting, and centrifugal casting. The other processes mentioned in the introduction are still in developing stage.

A. Dry Powder Processing

Here the technique is used to produce functionally graded material through three basic steps namely: weighing and mixing of powder according to the pre-designed spatial distribution as dictated by the functional requirement, stacking and ramming of the premixed powders, and finally sintering as shown in Fig.2. This technique gives rise to a stepwise structure. Starting from the powder material it is possible to obtain nearly optimal conditions for graded materials varying in composition and microstructure. This performed by changing the chemical composition or average particle size of the applied powders. If continuous structure is desired, then centrifugal method is used. The powder metallurgy method is one of the most commonly employed techniques due to its wide range control on composition, microstructure and shape forming capability [3].

Mahmoud M. Nemat – alla et al. used steel/aluminium FGM with composition changing from 100% steel in one side to 100% aluminium in the other side. In their fabrication they found that the sintering temperature should not exceed 600°C, if it so a new component will be formed. Also increasing the number of layers in steel/aluminum FGM can decrease the sharp interface between the layers and produce FGM instead of functionally graded layers material. The fabricated steel/aluminum graded material specimen with very smooth transition will leads to disappearing of the thermal stresses singularities and minimizing the stress concentration values [4]. Xin jin et al. fabricated eleven layered ZrO_2/NiCr FGM in which the matrix phase varies from the metal to the ceramic and the inclusion phase varies from the ceramic to the metal. With the increase of ZrO_2, the hardness increases tediously and the ductility decreases gradually. Here the ceramic matrix provides the rigid skeleton and constrain the plastic flow of the metal.

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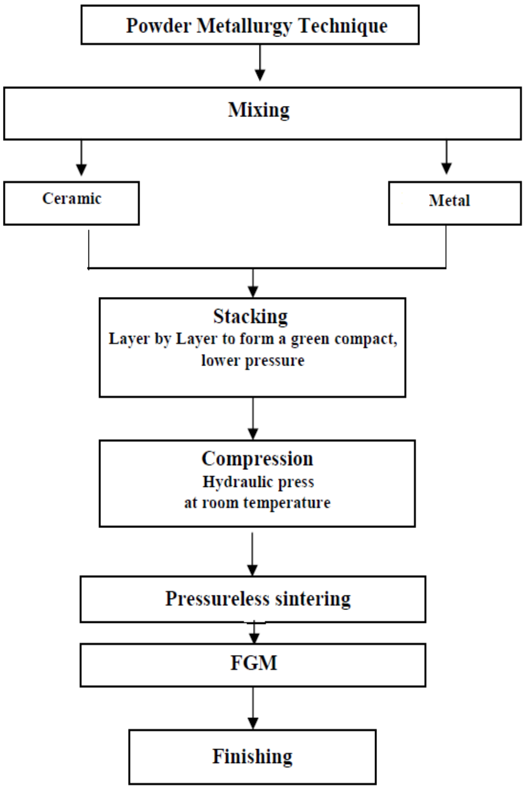

Figure 2. Flow chart of powder metallurgy technique for producing FGMs

The bending strength and elastic modulus firstly decrease as the volume fraction of ZrO_2 increase from 0% to 50%, and then increase as the volume fraction of ZrO_2 increase from 50% to 100%. These are mainly affected by the weakly bonded ceramic/metal interface. And the porosity does not seem to have an obvious effect on the distributions of the mechanical properties in the ZrO_2/NiCr FGMs [5].

B. Slip Casting

The slip casting is a powder based shaping method traditionally applied in the ceramic industry. In general slip casting is a filtration process where powder suspension is poured in a porous plaster mould. Due to the resulting capillary forces the liquid is removed from the suspension (slip) and the powder particles are forced towards the walls. A gradient will be formed by changing composition or grain size of the applied powder suspension during the slip casting procedure. This technique also requires subsequent consolidation step, where the powder is densified (sintered) and a gradient structure of the FGM results [6]. Tomoyuki Katayama et al. fabricated a functionally graded material (FGM) from tungsten and alumina powders. Two types of W powder, with different oxidizing properties, were used as the raw powders for the Al_2O_3–W FGM. “Oxidized W” was prepared by heat treatment at 200 °C for 180 min in air. The green compacts were subsequently dried, and then sintered using a vacuum furnace at 1600 °C for a fixed time. The green compact obtained with the as-received W powder showed a clear interface between Al_2O_3 and W as a result of the huge difference between the densities of the powders. However, with the oxidized W powder, the green compact revealed a W particle distribution which gradually varied, resulting in a microscopic compositional gradient [6].

C. Centrifugal Casting

Centrifugal force can be used to create a gradient composition in a metallic melt that contains another solid phase. Generally, fabrication of FGMs by the centrifugal method is classified into two categories based on the melting temperature of the reinforcement particle. If the melting point is significantly higher than the processing temperature, the reinforcement particle remains solid in a liquid matrix.

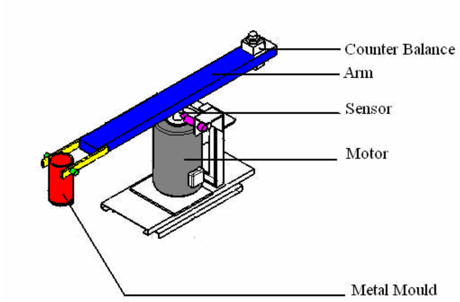

Figure 3. Vertical centrifugal casting setup

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This method is named as a centrifugal solid-particle method (CSPM). The selective reinforcement of the component surface obtained by CSPM results in a higher wear resistance in the outer surface as well as maintaining high bulk toughness. On the other hand, if the melting point of the reinforcement particle is lower than the processing temperature, centrifugal force can be applied during the solidification both to the reinforcement particle and to the matrix. This solidification is similar to the production of in situ composites using the crystallization phenomena, and this method is, therefore, named as a centrifugal in situ method (CISM). The formation mechanism of the compositional gradient during the fabrication of FGM by the centrifugal in-situ method in the A–B alloy is, 1) Partial separation of A and B elements in the liquid state occurs due to the density difference. 2) A compositional gradient is formed before the crystallization of the primary crystal. 3) The primary crystals in the matrix appear according to local chemical composition. 4) The primary crystals migrate because of density difference, and a further compositional gradient is formed [7].

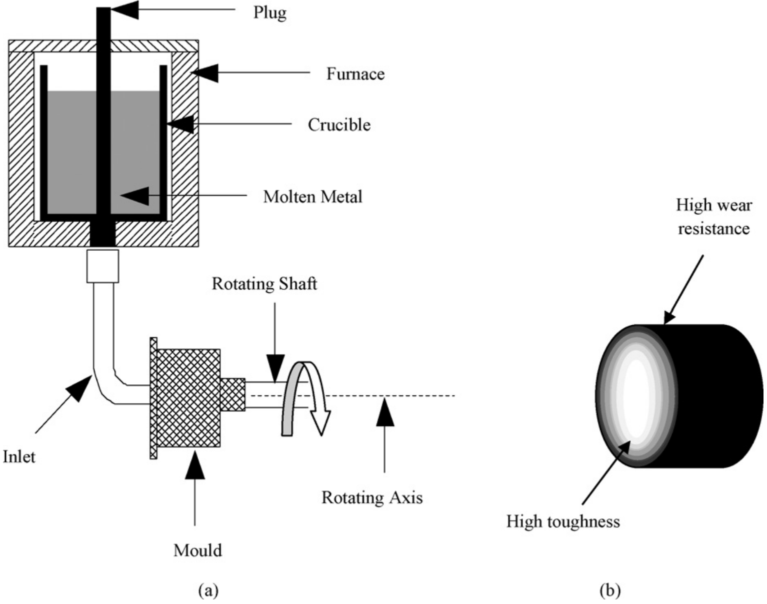

Figure 4. Schematic representation of: (a) the horizontal centrifugal casting process and (b) the final product obtained (Al - High toughness inner region and SiC – High wear resistance)

Usually, the density of the outer part of the FGM rings fabricated by the centrifugal in situ method is larger than that of the inner part of the ring. In general, steeper compositional gradient appears for the CSPM. Since the motion of the solid-particles under the centrifugal force is governed by stoke's law (Migration distance is greater for large particles). It is well known that, the mechanical properties depend on particle size distribution as well as the volume fraction of particles in particle reinforced or dispersion strengthened of any composite material. From the research works it is found that the extent of particle segregation and relative location of enriched and depleted particle zones within the casting are mainly dictated by the relative densities of the particle and liquid, teeming temperature, melt viscosity, cooling rate, particle size, solidification time and magnitude of centrifugal acceleration [8]. G. Chrita et al. studied the effect of centrifugal casting technique on castings as compared to the traditional gravity casting. They concluded that the centrifugal effect may produce an increase in rupture strength by 50%, rupture strain by 300% and young's modulus by 20%. Also an important and interesting finding in their study was, higher the distance in relation to the rotation centre (higher centrifugal force) the bigger the increase in mechanical properties. According to their study, the centrifugal effect on castings may be divided in three main features: centrifugal pressure, intrinsic vibration of the process, and fluid dynamics. The effect of each of these variables will be the responsible for the differences in both mechanical and/or metallurgical properties on the castings [9]. Yoshimi watanabe et al. conducted their examination to determine the particle size distributions in the FGM tubes were fabricated from plaster/corundum model materials by centrifugal solid - particle method. During solidification the reinforcement position is heavily depends on the 'G' number. Centrifugal force magnitude 'G' is given by,

$$G = \left(\frac{\omega^2 \times r}{g}\right) \qquad (1)$$

Where 'R' is the radius of the arm in meters, 'ω' is the arm rotational speed in rad/sec and 'g' is the acceleration due to gravity. In here they considered three levels of centrifugal force listed as 15, 28 and 45 with five different corundum particle sizes for the fabrication of FGM cast tube. The particle size gradient in the FGM becomes steeper by increasing the 'G' number or by decreasing the mean volume fraction of particles [10].

In an study of Yoshimi watanabe et al. commercially available Mg alloy, ZK60A was used as a mater alloy to fabricate Mg based FGM with a mould temperature of 680°C with a cooling rate of 0.05°C/s. Results of EDX analysis showed that Zn concentration at each region was almost the same i.e weak peaks at middle and inner region, while Zr exists only in the outer region as strong peaks. Graded distribution of Zr phase is enhanced with increasing the 'G' number. Finally these graded structures are caused by the difference in the formation mechanisms of compositional gradient during the centrifugal method between Zr and Zn, since the solid Zr particles exist prior to the application of the centrifugal force. In case of hardness phenomena the maximum hardness value was attained at outer strong Zr phase region and it increased in the direction of centrifugal force [7]. Al-Al_2Cu functionally graded material (FGM) ring was fabricated from Al-3 mass%Cu initial master alloy by the centrifugal in-situ method. In the case of Al-3 mass%Cu alloy, the density of the primary α-Al crystal is larger than that of the molten Al alloy. Therefore, the solid α-Al phase migrates towards the outer periphery of the ring when the centrifugal force is applied in the early stage of solidification. Consequently, since the Cu concentration within the FGM ring monolithically increases towards the ring's inner position, the FGM ring, whose density increases toward inner region, can be successfully fabricated by the centrifugal in-situ method from dilute Al-Cu alloy. It is also found that the hardness increases towards the inner region of the ring within the Al-Al_2Cu FGM ring [8]. A.S.Kiran et al. used centrifuge processing technique for the Al-Si FGM fabrication with consideration of the processing parameters like teeming temperature (900°C) and rotational speed of mould (400rpm). In the solidification primary Si particles moved in the direction opposite to the direction of the 'G' force as the density is less than that of liquid aluminium. Brinell hardness test shown that, the hardness is mainly affected by the content of primary Si content along the polished specimen [11].

Shimaa El-hadad et al. were fabricated Al-5mass%Zr functionally graded materials by centrifugal solid – particle method under applied centrifugal force of 30, 60 and 120G. Microstructural observation along the centrifugal force direction showed that Al_3Zr particles are almost oriented normal to the applied centrifugal force direction. Also increasing centrifugal force resulted in a steep particles distribution and decreased thickness of the intermetallics rich area. From the block-on-disk wear tests the anisotropy of wear property of the current Al/Al_3Zr FGMs was diminished with decreasing the applied centrifugal force and thence decreasing the particle orientation. An enhanced wear resistance was achieved in the Al/Al_3Zr FGMs by controlling the distribution of both the orientation and the volume fraction of Al_3Zr particles. Also they concluded plastic deformation induced wear was the dominant wear mechanism [12].

Xiaoyu huang et al. were fabricated Al-Si alloy based composite pistons reinforced with SiC particles locally at the head by centrifugal casting with parameters consideration as slurry temperature of the alloy, the mould temperature and the rotational speed of the mould on the particle segregation. By the SEM analysis, it is clear that a large quantity of SiC particles are in the piston head, which meets requirements such as hardness, wear resistance and thermal expansion behavior of pistons. The hardness values along the axis of piston gradually increased from the skirt to the head, which corresponds to the structure changing along the axis of the pistons. At a slurry temperature of 850°C, a mould temperature of 600°C and a rotation speed of 800rpm possessed the highest value of hardness with best wear resistance when compared with the piston fabricated by permanent gravity mould casting [13]. H.P Thirtha Prasad et al. used unidirectional solidification mould made up of graphite with the process considerations as slurry temperature of 750°C, rotational speed of the mould as 200rpm and maximum centrifugal acceleration of 54g for the fabrication of Al/ Al_2O_3 FGM. From their microstructural evaluation, most of the Al_2O_3 particles enriched in the external zone of the specimen under centrifugal force, and some congregated Al_2O_3 particles with low bulk density segregate to the inner reinforced zone of the cylinder. The mechanical properties of FGM were increased with increasing Al_2O_3 particles. Also the fracture surface of the FGM at the center was ductile nature and the outer was brittle nature [14]. Li chanyun et al. carried out hydraulic simulation experiments on vertical centrifugal casting machine with two different filling methods (top filling and bottom filling) and three kinds of rotational velocities (163, 245, and 375rpm) to determine the best filling method. A high speed camera photos taken in the experiment showed that in both top filling and bottom filling, liquids stick to back-wall of cavity or runner to filling due to the action of centrifugal force. Filling volume is rising with the increase of the filling time and rotational velocity of mold. Experiments on titanium alloy resulted that the bottom filling method is better than the top one, which can achieve stable filling, minimize turbulence and avoid drastic liquid collision [15].

R. Sivakumar et al. carried out their investigation on mullite-molybdenum graded cylinders by centrifugal molding technique with ceramic rich in inner surface and metal rich in outer surface were fabricated with the smooth increase of Mo content toward outer direction. Microstructural observation and EDX analysis performed showed linear gradation of Mo content along the radial direction of cylinders. The microstructures of graded hollow cylinders reveal that the methodology of slurry preparation has a vital influence on the gradation of Mo along the radial direction. Measured Vickers hardness values proved the continuous compositional change from inner to outer surface of graded cylinders and the hardness became constant in outer surface of graded specimens due to formation of interconnected Mo structures [16]. J.W. Gao et al. investigated the solidification process during the centrifugal casting of FGMs numerically and validated results against the experimental results. During solidification, the particles move to the outer or inner direction of the mould under centrifugal field depending on the particle density relative to that of the melt. Solidification is induced from the outer wall of the mould by convective cooling while the inner wall is assumed to be adiabatic. The model was used to investigate the solidification process in centrifugal casting of Al/SiC FGMs in a cylindrical mould. Three factors can be identified to be responsible for creation of the particle concentration gradient: the geometrical nature of particle flow in the cylindrical mould, the angular velocity, and the solidification rate, which captures the desired gradient — it is the interruption of particle migration by the solidification front that creates gradients in the particle concentration. By optimizing processing conditions, such as the particle size, initial particle concentration, rotational speed of the mould, cooling rate and superheat, one can engineer a desired gradient in the solidified part [17]. A.C. Vieira et al. have presented wear characteristics of Al/SiC_p FGM fabricated by centrifugal casting process with two different mould rotating speeds (1500 and 2000rpm). Under a constant acceleration, the velocity (V) of a spherical particle of size (R_p) may be estimated using stoke's law by,

$$v = \frac{2{R_p}^2(\rho_p - \rho_l)\gamma}{9\eta} \qquad (2)$$

Where, ρ_p and ρ_l are the densities of the particle and liquid and η are the viscosity of the liquid. From Eq. (2) it can be seen that a higher centrifugal force (acceleration) will result in a higher particle velocity, emphasizing the reinforcement gradient along the centrifugal direction. FGM cast at low centrifugal speed (1500 rpm) presented a smooth gradient on SiCp distribution, while FGM cast at higher centrifugal speed (2000 rpm) revealed a sharper gradient on the distribution of reinforcing particles. This gradient was controlled by the movement of the solidification front, blocking the mobility of SiC particles in the melt. For the aluminium based

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FGM composites considered in this study, two-body abrasion wear, oxidative wear, adhesion and delamination were the main wear mechanisms identified [18].

D. Tape Casting

The tape casting process is shown in Fig.5. A slip, powder containing suspension is distributed on a carrier film in a thin casting type. The cast tape thickness is generally in the range of 25µm to 1mm. Minimal tapes down to 1µm could be produced.

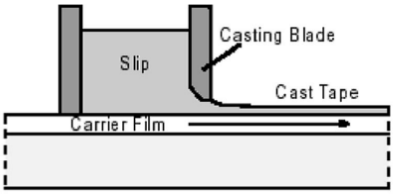

Figure 5. A tape casting process setup.

Different stages of tape casting are, 1) developing slip which contains water, powder particles and binder, 2) drying of green body or tape, 3) consolidating of dense material of tape. In order to achieve FGM, tapes of different composites were prepared. Square units were cut of the green tapes which were subsequently placed on top of each other. The thickness of the applied tapes is in the range of 200µm. The densification of tape is achieved by sintering. Stepped gradients of metal-ceramic and ceramic-ceramic materials are produced by casting of tapes of different composition and subsequent lamination. Anne-Laure Dumont et al. fabricated MoSi2/ Al_2O_3 FGM with alumina contents varying from 20 to 80 mol% using a combination of tape casting and self-propagating high-temperature synthesis (SHS). After debinding, the green samples were ignited at room temperature. The combustion reactions were conducted under a weak load to enhance the densification of the composition-graded composites. The porosity of the multilayer samples is significantly reduced when a low pressure (typically 3MPa) is applied during the SHS stage and when the green layer thickness is greater than 500 mm. The conservation of the multilayer structure is strongly dependent on the control of the amount of liquid formed during the SHS reaction [19]. Kongjun Zhu et al. were developed a piezoelectric actuator from the graded composition of PbO, Nb_2O_5, ZrO_2 and MgO powders. Results showed that sintering temperature has inverse effect in porosity and positive relation with the grain settlement. A non uniform grain size induces poor density of the ceramic [20].

III. APPLICATION OF FUNCTIONALLY GRADED MATERIALS

Some of the applications of functionally graded materials are discussed below.

A. In Aerospace and Automotives

Space Shuttle utilizes ceramic tiles as thermal protection from heat generated during re-entry into the Earth's atmosphere. However, these tiles are prone to cracking at the tile / superstructure interface due to differences in thermal expansion coefficients. An FGM made of ceramic and metal can provide the thermal protection and load carrying capability in one material thus eliminating the problem of cracked tiles found on the Space Shuttle. The interest in graded materials like Aluminium/Silicon carbide (ceramic – metal) are focused primarily on the control of thermal stresses in elements exposed to high temperatures (to 1600°C) for instance aerospace structures and Cu/SiC for dynamic seal applications.

Thermal Barriers Coatings made up of ZrO_2 and NiCoCrAlY FGMs are very popular as thin layers protecting of aircraft engine components against the thermal shock (e.g. turbine blades). Boron additions to conventional titanium alloys have the potential to form lightweight, high modulus, dispersion strengthened, discontinuous-reinforced composite material structures enabling replacement for significantly more dense steel and nickel materials.

Exhaust and propulsion systems (e.g. Thrusters) that are required material properties like enhanced resistance to high temperature, thermal shocks, wear, oxidation and corrosion. Power transmission systems (e.g. Valves) that are required material properties like low specific weight, resistance to high temperature, resistance to wear and corrosion. Braking systems (e.g. Brake disks) that are required material properties like resistance to high temperature, friction and wear, high bending strength in room temperature and high temperature regimes, enhanced thermal conductivity, structural stability in temperature cycles, lower specific weight.

B. In medicine

In dental implantation a more realistic FGM is usually composed of collagen hydroxyapatite (HAP) and titanium is used [21]. Porous hydroxyapatite (HA) scaffolds with a functionally graded core/shell structure was fabricated for biomedical applications [22]. TiO_2 has also been used in combination with hydroxyapatite for developing biomaterials for implants because of its favourable biological effects and improved corrosion resistance. TiO_2 is also frequently and successfully used to reinforce Al_2O_3 wear resistant coating on metal substrate [23].

C. In defence

One of the most important characteristics of functionally graded material is the ability to inhibit crack propagation. Metal ceramic FGMs used in structures as fire retardant doors and penetration resistant materials for armour plates and bullet-proof vests. One of the available material compositions gradually shifts from titanium diboride to a combination of titanium and titanium diboride, combining the ceramic's ability to absorb energy with the toughness of a metal - ideal for vehicle armor solutions [22].

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D. In Optoelectronics

Piezoelectric and thermoelectric devices, high density magnetic recording media, in optical applications as graded refractive index materials in audio-video discs.

E. In Industry

A ceramic roller suitable for various hybrid bearing applications in industries made up of silicon nitride as base material. This material possesses excellent mechanical properties, ideal for load bearing applications. In many cases, entire components are made of tungsten carbide, for an application requiring wear resistant properties. With a gradient, the use of expensive carbides can be minimized; thus lowering the total cost of the component. Functionally graded metal matrix composites (FGMMCs), especially gradient particulate composites with aluminum matrix, have been used in important applications such as in electronic packaging industry, for brake rotor assemblies in automobile industry and as armor materials [24].

IV. FUTURE SCOPE

Many research works carried out in the metal-ceramic FGMs such as (Al/ Al_2O_3, WC/Co, Mo/Mo_2C, Ni/ Al_2O_3, ZrO_2/NiCr and WC/Ni) for their proposed application. Based on the research history it is clear that the FGM processing stated with the ceramic compounds. Up to now so many processing technologies were discussed on the metal-ceramic type for the applications like which required high temperature resistance and toughness with light weight material. In contrast metal-metal combination having only least research works it may be due to the simplicity in the production processes of ceramic-ceramic and metal-ceramic combination. With the consideration of industrial needs new FGM families and their processing methods are to be developed especially with respect to real applications. In the field of FGM gradation forming is the critical step. To overcome this computer assisted modelling should be developed to improve forecasts for the proper gradient formation. Calculations related to the properties and combinations of the desire FGM should be done prior to the production. After the production of FGM, characterization is to be carried out by new testing methods in a non destructive way. Up scaling of laboratory route into industrial scale is necessary. In this regard a lot of works still needed to adopt the existing processing routes to industrial viable production routes. Industries are based on cost effective processes for the production either it may be mass or low volume. So technologies which transfer the FGM from laboratory to industry are to be invited. Also reproducibility in geometry, gradation and property of a FGM is very much important this has to be attained.

V. CONCLUSION

Functionally graded materials are very important in engineering and other applications which requires special properties which are not satisfied by the conventional materials like naturally available materials, alloys and metal matrix composites. Also we have discussed so many processing routes for making them to the special applications. In this regard here we present some of the deficiencies related to each processing technique for the further improvement. Commonly all the processing routes start with powders of base. During the consolidation for the gradient forming step, due to the lack in management of sintering temperature and time distortion in the final part arises with unequal density. To overcome this, a prior sintering step is to be implemented before the production in order to reach the desired part of final density.

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