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Review

A review on the fabrication techniques of functionally graded ceramic-metallic materials in advanced composites

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Whenever two or more dissimilar materials are bonded, large jump in the stresses occurred at the interface during the fabrication will leads to the delamination and poor load-bearing performance on the final structures. Functionally graded material (FGM) which has compositional and microstructure gradient along its thickness was introduced to be a great solution to this problem. Aiming for the best technique to be implemented, this paper presents an extensive review on the various fabrication techniques of FGMs composed by metallic and ceramic phases. Fabrication techniques in this field of work have incorporated many concepts from different background of gradation processes and consolidation or sintering processes. Each of these processes however has their own advantages and disadvantages. The best technique to be applied can be found by considering some critical issues highlighted in published literatures. This review concluded the powder metallurgy (PM) as the most suitable technique certainly for mass production and up-scaling of the FGMs. The selection was strengthen after considering the advantages of the technique such as process cost-effectiveness, reliability of the practical implementation of the process and the high capability of the process to control the quality of the FGMs.

Key words: Functionally graded material (FGM), fabrication, powder metallurgy (PM).

INTRODUCTION

Functionally graded materials (FGMs) were introduced as a class of advanced composite that composed of more than single continuous or discontinuous gradient in composition and microstructure. These materials have a flexibility in term of the behavior as the property of one side of the FGMs is differs from the other side. One side of the FGMs structures could have higher mechanical strength and thermal resistance while the other side has different worth on the property. Position-dependent chemical composition, microstructure or atomic orders are among the factors that influence the property gradient in the materials. By controlling all these factors, the property gradient could be customized to meet any specific need within the best utilization of the composite components (Lannutti, 1994; Shukla et al., 2007).

Many theoretical works on designing and investigating

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the performance of FGMs were reported since 1970's but unfortunately, the impact of the outcomes was still limited until present time due to a lack of suitable fabrication method for the FGMs (Bever and Duwez, 1972; Shen and Bever, 1972). The most applicable method for the fabrication of FGMs is difficult to be defined because each method may shows disadvantages instead of some advantages. Elaborating the preparation of FGMs, the process basically consists of two steps which are known as gradation and consolidation. In each of these two steps, there are some different kinds of processing types. Such as for the gradation, the process could be performed whether by consecutive, homogenizing or segregating steps. Consecutive process depends on a stepwise gradient built-up of the structure from powder materials; homogenizing process shows a sharp interface between two materials is converted into a single gradient by a material transport and another type which is segregating process indicates the conversion of a macroscopically homogeneous material into a single gradient also by material transport that is caused by an external field such as gravitational or electric field.

According to the theory, the first stage in preparing FGMs is making the gradient and the process known as gradation process. Gradation process using PM method can be done based on chemical composition of elemental powders, porosity or pore-size, volume fraction of the phases and the particle size. It can be said that the gradation process is actually independent to the consolidation process. No matter how the gradation of the structure is made, the consolidation process becomes another issue. In the sintering stage, the bonding of the graded layers is the main consideration and it is strongly dependent on the sintering process of the structure. In order to successfully fabricate the FGMs specimens, researchers have applied the optimal combination of several methods depending on the properties of the component materials (Kiebeck et al., 2003; Miyamoto et al., 1999; Li et al., 2003).

PROCESSING TECHNIQUES OF FUNCTIONALLY GRADED MATERIALS (FGMs)

There are several different physical and chemical methods depending on type of materials, potential application and available facilities for the FGMs fabrication (Schwartz, 2002). The most updated FGMs fabrication methods proposed by researchers in the related field are discussed in this part of the work. Basically the techniques are classified into two different techniques named constructive and transport based processes where each of them is composed of various sub-steps that need to be followed to complete the fabrication. By implementing constructive process which allows full and potentially automated control of compositional gradients, the gradation process is made by stacking more than one starting materials selectively until layered structure is produced.

The advantage of this technique is that it is able to produce unlimited number of gradient. The situation will be different for the case to make gradation from the flow of fluids, the diffusion of atomic species and the conduction of heats. The transport based technique is the appropriate method for such cases as the case utilizes natural transport phenomena to create compositional and microstructural gradients during fabrication of the FGMs. The existing and most updated techniques for FGMs fabrication will be discussed in the following part of the work.

Powder metallurgy

PM is an apparent technology for the FGMs fabrication and is increasingly being used to create gradients on material. This method is appropriate for FGMs fabrication using solid materials. In PM route, some steps are needed for the completion of the product preparation. These steps can be classified into four main steps namely: powder preparation, powder processing (weighing and mixing of powder according to desired percentage of composition), forming operations (stacking and ramming of premixed powders) and finally sintering or pressure assist hot consolidation. After completing the sintering process, optional secondary processing can be performed to enhance the performance of the structure.

Several techniques have been introduced for powder preparation such as through chemical reactions, electrolytic deposition, grinding or comminution. These techniques permit mass production rates of powder form materials and it usually offered within controllable size range of the final grain population. For the powder processing, the main consideration is focused on the precision in weighing amounts and the dispersion of the mixed powders. These elements will influence the structure properties and should be handled in very careful way. In the subsequent processes, the forming operations is performed at room temperature while sintering is conducted at atmospheric pressure as the elevated-temperature used may cause other reaction that may affected the materials. At this stage, the atmosphere condition must be appropriate since high-temperature process has high sensitivity to the surroundings.

Hot pressing

Yittria stabilized zirconia (YSZ) and nickel 20 chromium (NiCr) are the two materials combined using YSZ-NiCr FGM interlayer via hot pressing method (Li et al., 2003). At the initial stage of the processing, the powdered YSZ and NiCr were mixed in ball milling machine for 12 h before being stacked layer by layer in graphite die coated

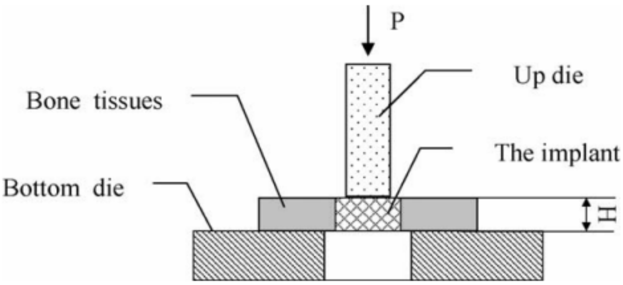

Figure 1. Schematic diagram for testing the shear bonding strength between the tissues and the implant (*P*: extruding force, *H*: thickness of implant) (Chu et al., 2006).

with boron nitride. This study applied the concept of stepwise gradation by arranging the composition of each layer to be in certain desired percentage. The pre-compaction of each layer was performed under a lower pressure before stocking the adjacent layer under higher pressure (10 MPa) to ensure the exact compositional distribution within the layers. The joining of YSZ-NiCr that performed at 1200°C for 1 h in vacuum of 5×10^{-5}torr atmosphere shows good thermal stability and oxidation resistance up to 1000°C before the crack initiate aggregately in the YSZ-rich NiCr75%vol.%YSZ interlayer during the thermal cycling to 1000°C. After 30 thermal cycles, the shear strength in NiCr-50%YSZ and NiCr-75%YSZ interlayers were found to be 207 and 75 MPa, respectively.

The crack propagation on the structure can also be benefited for other purpose such as reduction of FGM layers. This is made by the assistance of finite element modeling of thermal residual stresses of the FGM model which is verified by comparing the analytically calculated values with the measured values of the fabricated specimens. The crack on the FGM caused by the residual thermal stresses is possible to be estimated using maximum principal stress theory and maximum tensile stress theory. In the reduction of Si_3N_4-Al_2O_3 FGM layers using 3-D finite element modeling, the calculated maximum stress was found to be first principal stress of 430 MPa around 90%12H/10%Al_2O_3 area. Within a customized sintering profile for the fabrication, a new composition profile of 15 layers with crack free joint of the FGM was proposed as the optimized structure (Lee et al., 2008). Instead of the ceramics FGM, a bulk SiC/C FGM is another pair that is successfully fabricated using hot pressing process. In term of thermal properties, the hot-pressed SiC/C FGM was found having high effective thermal conductivity at the interface of 1 mm SiC layer when compared to the specimens prepared using other methods. No cracks were found on the SiC/C coatings, thus make the FGM possess high thermal fatigue behavior. The plasma-relevant performance also indicated that the specimen has excellent high-temperature erosion resistance (Wu et al., 2005).

The other properties of FGM which is in term of comprehensive biocompatibility and bonding strength with bone tissue in the rabbit were investigated by *in vivo* studies (Chu et al., 2006). Same as applied previously, 12 h ball milled HA/Ti powders were stacked layer by layer according to the pre-designed compositional profile of Ti/Ti-20vol.% HA/Ti-40vol.%HA FGM before all were compacted together under 200 MPa. The hot pressing is applied after that, at 1100°C under pressure of 20 MPa in nitrogen atmosphere for 30 min with a heating rate and cooling rate of 10°C and 6°C/min, respectively. Figure 1 shows the schematic diagram for testing of the shear bonding strength between the fabricated FGM and human bone tissue.

The shear fracture for Ti-40vol.%HA graded layer in the FGM implants occurred in new bone tissues zones near the interface between the FGM implants and bone tissue after three months *in vivo* period. The study concluded that the HA/Ti FGM is highly potential for the application in hard tissue replacement as it possess high bonding strength which could exceed the 4.73 MPs shear strength of new bone tissues compared to pure Ti metal. The bonding strength between gold alloy and porcelain fabricated via hot pressing has been optimized in a study which evaluate the influence of a composite interlayer on the shear bond strength of a metal-ceramic composite when compared to a conventional porcelain fused to metal (PFM) (Henriques et al., 2011). The study found that the shear bond strength for all composites joined to metal and to ceramic substrates were higher (>150 MPa) than the upper range of conventional PFM techniques which is about 80 MPa. The use of functionally graded composite interlayer also proved enhancing the metal/ceramic adhesion in 160%.

Cold pressing

A beam-shaped porous lead zirconatetitanate-alumina (PZT-Al_2O_3) FGM actuator that exhibits the theoretically matched electric-mechanical response with crack-free structure built based on the pyrolyzable pore-forming agent (PFA) porosity gradient has been successfully fabricated using cold sintering method (Li et al., 2003). The powder mixture of PZT, Al_2O_3 and the stearic acid as the PFA were initially stacked in a die at 100 MPa and restack using cold isostatic pressing (CIP) before the normal sintering process at 1473 K temperature for 1 h takes place. The binder addition is the same thing applied in another fabrication of FGM composed by Ni and Al_2O_3 which is in purpose to investigate the influence of the particle size used. In this study, the appropriate Ni, Al_2O_3 and Q-PAC 40 (organic binder) particle sizes were selected based on the desired microstructure at the corresponding composition. After mixed together through blending process, the powder mixtures were cold pressed under 86 MPa pressure. The process followed by pressureless sintering at 1350°C with specific sintering

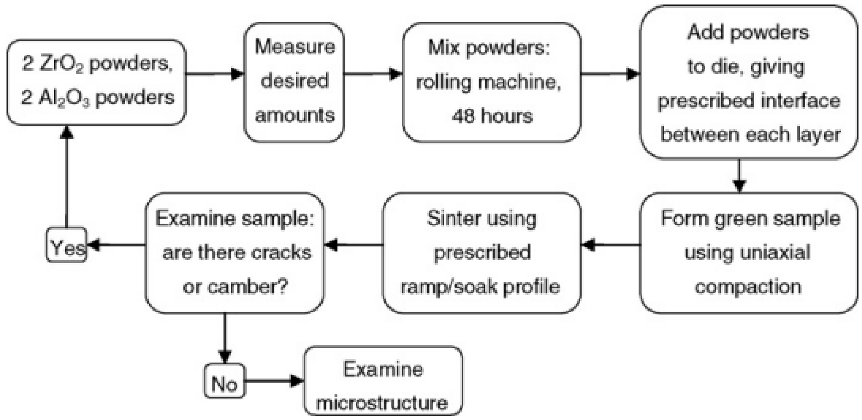

Figure 2. Flow chart detailing the manufacturing process of Al_2O_3/ZrO_2 FGM used in Sun et al. (2008).

cycle which is developed based on the microstructure conditions at every soaking time (Michael and Hugh, 2006). The titanium/hydroxyapatite (HA/Ti) and other FGM implants with gradually change composition in the longitudinal direction of cylindrical shape were also fabricated via cold isostatic pressing (800 to 1000 MPa) to optimize the mechanical and biocompatibility properties of the structures. Different specimens sintered via three different methods were evaluated to see the effect of the sintering method on the stress relaxation in the implanted region of bone.

The result found that spark plasma sintering method is the best to be applied for the production of better stress relaxation in the FGM structure as the Brinel hardness measurement of the specimen shows the smoothest gradually decreasing values. The gradual reaction occurred on the tissue in response to the graded structure of FGM were demonstrated and it implies the possibility to control the tissue response through the gradient function of FGM (Watari et al., 1997). For the other issue of fabrication, two main problems in preparation of particles reinforce-ment associated with porosity and agglomeration FGMs were highlighted as the smaller shrinkage can lead to higher thermal residual stresses while the reduced consolidation will cause weaker mechanical properties to resist the stresses. Relating the fabrication method with the thermal residual stress, it is reported that there is no way to match the compaction and sintering behavior of two dissimilar materials while making co-sintering of multi-layered FGMs (Cai et al., 1997).

Therefore, the idea on manipulating the powder characteristics by mixing powders with distinct properties was proposed to overcome the problem. The factor of powder characteristics are being controlled because one of the characters which is the mismatch in coefficient of thermal expansion was found influencing the residual thermal stresses occurred within the FGMs during fabrication. Instead of that, the optimum processing parameters for fabrication of Al_2O_3-ZrO_2 (alumina-zirconia) FGM has been obtained by changing the compacting parameter ranged from 60 to 210 MPa, and the cooling/heating rate at several ramp/soak sintering cycle (Sun et al., 2008). Figure 2 shows the flow chart of the manufacturing process of the Al_2O_3-ZrO_2 FGM used in this study. Different elemental consideration under powder characteristic that is in term of the addition of the space holder material was investigated on porous Ti-Mg (titanium-magnesium) FGM.

The fabrication process started with the mixing of the Ti, Mg and the NH_4HCO_3 (ammonium hydrogen carbonate) as the space holder agent in a ball milling machine, followed by cold compaction of the powder mixtures using hydraulic press machine at 550 MPa and two heat treatment steps. In the first heat treatment step, the space holder agent was burnt out by heating the powders at 80℃ for 4 h before the temperature is increased to 110℃ for 2 h to remove the residual moisture from the specimen. The fabrication process is complete only after the sintering process by heating the structure up to 630℃ for 2 h was performed. To prevent the oxidation, all the processes were performed at argon atmosphere. The study concluded that the amount and particle size of the space holder material do affected the porosity, phase constitution and compressive strength of the Ti-Mg FGM.

Characterization on the microstructure and the mechanical properties of the fabricated Ti-Mg FGM showed indicate good performance of the structure with porosity ranged from 30.8 to 54.8%, 100 to 400 µm macro pore size, 27.2 to 139.9 MPa compressive strength and 1.26 to 4.01 GPa elastic modulus. As all these values close to those belong to human bone, the properties were considered acceptable for bone replacement application. In the most recent study, HA/Ti FGM cylindrical plate with 6 mm thickness and 20 mm diameter was fabricated via pressureless sintering process. The FGM plate was constructed from some layers composed by various mixing ratios (100:0, 75:25, 50:50, 25:75 and 0:100). The Vicker's hardness of the fabricated sample was found higher than that of pure micro crystalline Ti metal and reducing within the decreasing density of the FGM (Shahrjerdi et al., 2011).

Most researchers have more intention to use micro scale particles in the fabrication of FGMs since the nano size particles need more precision in the processing. Only small number or limited studies were reported using the nano size composition particles. Co/α-Al2O3 FGM composed of nano size powders was successfully fabricated using high pressure torsion procedure (Menéndez et al., 2008). This procedure is classified under PM method and cold pressing as the consolidation or sintering process is performed after the compaction. The different is only at the way of giving the pressure in torsional mode.

Sintering process

The gradation that has been performed in the powder compact needs to be preserved during sintering or consolidation process. Some of light metallic powders such as magnesium and aluminum will tend to react with the oxygen and disperse to the atmosphere in oxidation which should be avoided in order to get proper resulting materials. The sintering process is performed simultaneously with the compaction process if the FGM is prepared using hot pressing process. However in cold pressing process, the sintering process is performed only after the powders were compacted. The effectiveness of three different sintering methods including electric furnace heating, high frequency induction heating and spark plasma sintering (SPS) were investigated (Watari et al., 2003).

In the study, PM method was implemented for the fabrication of Hap/Ti FGM and some other FGM implants to optimized both mechanical properties and biocompatibilities or charge bio-reactivity in each region of the specimens. The SPS sintering method was found applicable for the fabrication of HAP/Ti FGM as it has successfully fabricated the FGM with Brinel hardness that decreased from 61 in Ti to 15 in HAP. The properties lead to stress relaxation in the implanted region of bone. Another issue in sintering process which is the surroundings atmosphere should be highly considered to produce high quality FGM. Vacuum sintering should be better than normal sintering but the comparison between both have yet been reported. HAP/Ti FGM is the example of the specimen that has been successfully fabricated under vacuum atmosphere (Marcelo et al., 2006).

In order to evaluate the sintering performances the porosity is one of the parameter that could be considered. For this purpose, some sintering models have been developed and analyzed. The study proved that the rate at which the porosity is directly related to the rate at which shrinkage occurs (Pines and Bruck, 2006a). The change in porosity and shrinkage in theoretically sintered nickel/alumina (Ni/Al_2O_3) FGM have been studied (Pines and Bruck, 2006b). The study shows how the porosity reduction model can be used to access the quality of particle-reinforced metal-ceramic FGM formed by pressureless sintering and to predict changes that can be achieved in the porosity reduction through engineering of the particle dispersion for the processing of FGMs. The other sintering parameters including time, temperature, sintering atmosphere and the isostatic condensation influences on the performance of the resulting FGMs were investigated (Dobrzanski et al., 2011).

During the fabrication of the sintered tool gradient materials composed of wolfram carbide and cobalt used for the study, the sintering parameters were changed to find the optimum values. The sequential concentration of the molding with layers having increasing content of carbides and decreasing concentration of cobalt and sintering ensures the acquisition of the required properties including the resistance to cracking and abrasive wear of the tool gradient materials.

Infiltration process

Infiltration or the scientific term called hydrology is the process by which fluid on the ground surface precipitates into the soil. This process is governed by either gravity or capillary action forces. The rate of infiltration is depends on the soil characteristics such as storage capacity, transmission rate through the soil and the ease of entry. The rate and capacity of the infiltration process can be controlled by adjusting some parameters including the soil texture, vegetation types and cover, the content of water in the soil, the soil temperature and rainfall intensity. The infiltration method was introduced for certain FGMs with a complex shape preparation. The fabrication with this method needs little or no bulk shrinkage and more rapid reaction kinetics. As the common process for the mold shaping is heating the powder to a temperature that is higher than liquid phase, the requirement to make sure no bulk shrinkage is quite challenging.

From the literature, there is limited numbers of infiltration process implementation are reported. It is believed that the ticklish process handling and high costing demand are two main factors that make the infiltration become challenging to be used. The infiltration method has been implemented for FGM processing since many years before. A compositionally graded $Al\text{-}SiC_p$ composite was successfully fabricated using pressureless infiltration method at the early of last one

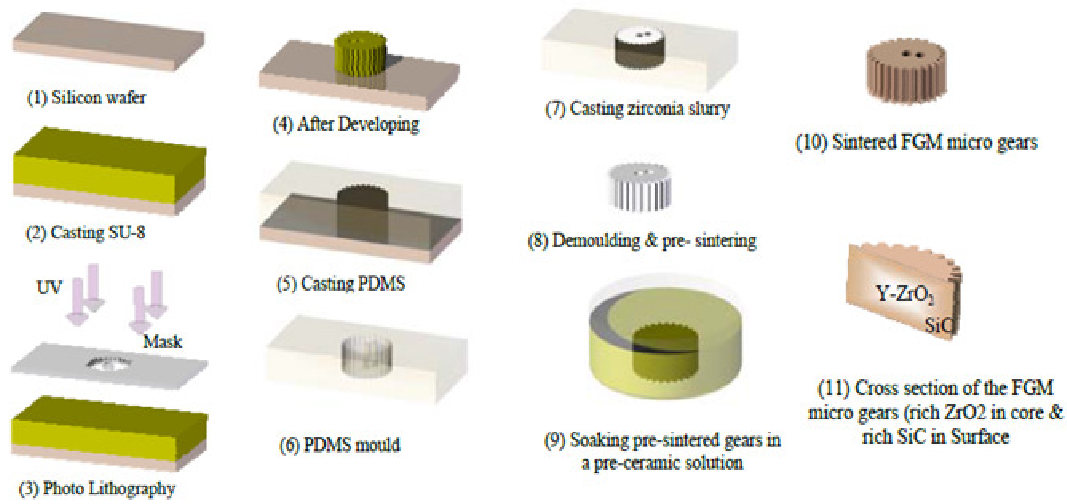

Figure 3. Schematic diagram of infiltration process of YSZ/SiC FGM (Hassannin and Jiang, 2010).

decade. It has been indicated that the thermal conductivity of the produced FGM increasing in non-linear trend while the volume fraction of the ceramic element decreased (Cho et al., 2004). An innovative way which introduced infiltration processing route with microwave sintering and environmental barrier coating (EBC) is subsequently presented for the fabrication of Si_3N_4 FGM that composed of α-Si_3N_4-Yb-silicate green parts and porous β-Si_3N_4 ceramics as the substrates (Willert-Porada et al., 2005).

The study found that the different green processing parameters and different heating methods were affecting the porosity of the pre-form materials. The other approach based on infiltration concept which developed certainly for the fabrication of micro ceramic FGM has been presented. Using this approach, the highly stable suspensions were carried out by controlling the colloidal behavior of the structures. It is highlighted that the highest possible stable suspension and demanded porosity of the FGM structure could be obtained by selecting certain pH and dispersant concentration during the fabrication. This technique (shown in Figure 3) proved that free standing micro components in YSZ/SiC FGM is possible to be fabricated via infiltration method (Hassannin and Jiang, 2010).

Different composition of porous Ti (pTi)/ hydroxyapatite (HA) FGMs were also fabricated using infiltration method which is performed in vacuum of less than 10^{-2} Pa and sintering at 1173 K for 7.2 ks. This study found that the porosity of the compacts is controllable in the range from 7 to 39% by the hot pressing temperature and applied load. The Young's Modulus of the fabricated FGMs was comparable to the human cortical bone in the porosity range from 24 to 34%. The study concluded that the Young's Modulus of the pTi can be customized by adjusting the content or volume fraction of the HA (Nomura et al., 2010).

The effects of some processing parameters on the performance of the infiltrated FGM were investigated, previously. The influence of particle size of composed materials on W-Cu FGM has been studied by evaluating two specimens fabricated using two different particle sizes. For liquid phase processing, this study concluded that the phenomenon of phase migration is driven by differential sinter ability in the gradient (Raharijaona et al., 2010). Besides that, the effect of glass infiltration was investigated on CaO-ZrO_2-SiO_2 system in the development of glass-alumina FGMs. In order to obtain the final compositional gradient which is indicated by blue glass, the glass formulation of the system was doped with cobalt by adding a small molar percentage (0.1 mol%) of CoO. The characterization of the specimens proved that the cobalt-doped glass have interesting mechanical properties including high elastic modulus, good fracture toughness and acceptable coefficient of thermal expansion (CTE). From the detailed micro-diffraction test made on the FGM cross section, the study revealed that the heat treatment and the glass porcelain not lead to any relevant crystallization of new phases after the process (Cannillo et al., 2008).

For other aspect, in order to investigate the effect of heating or sintering method, some different method which based on plasma facing factors including infiltration welding method, plasma spraying and resistance sintering under ultra-high pressure process have been used to fabricate W/Cu FGM (Zhou et al., 2007). This is the extended study of the infiltration-diffusion method efficiency which is previously implemented on W-C/brass FGM (Li et al., 2005). The analysis on the thermal properties of these specimens indicated that that element fabricated using infiltration-welding has the best thermal shock resistance compared to the others. The analytical

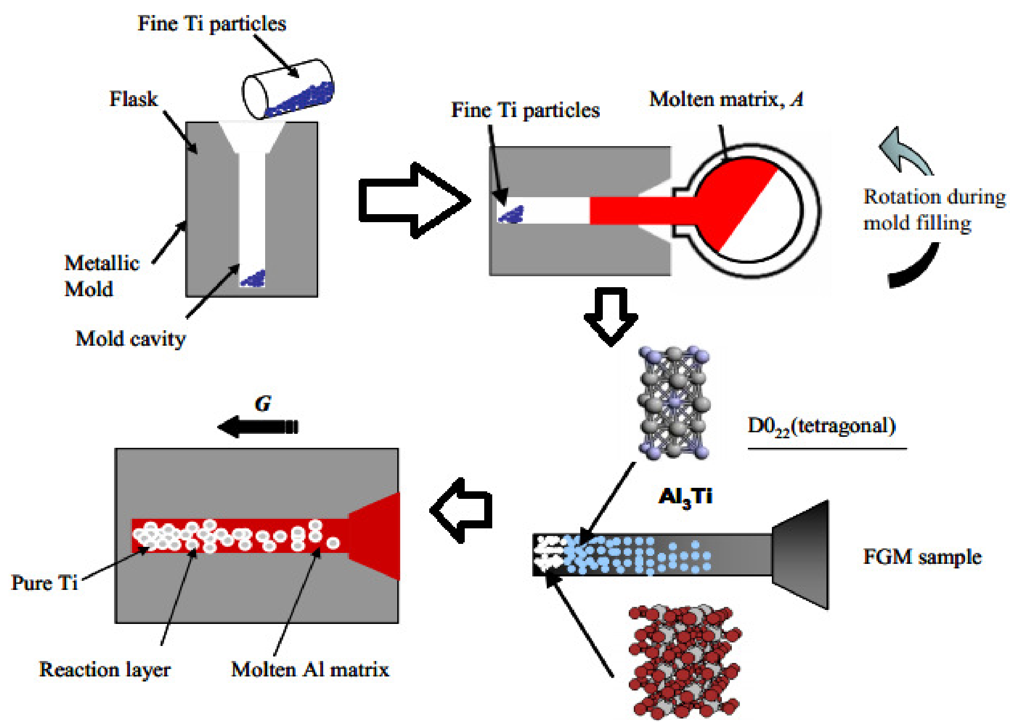

Figure 4. A schematic illustration showing CMPM (Watanabe et al., 2009).

analysis of residual thermal stress of FGM was performed on three types of $Al_{18}B_4O_{33}$/Mg FGMs which consist of different layer number and compositional gradient from 0 to 35%. The stresses were found decreasing in the macro-interface with the increasing layer number (Lee et al., 2009).

Graded casting process

Graded casting is another process for preparing FGMs with a complex shape. Basically clear definition about casting process is a manufacturing process by which a liquid material is poured into a hollow cavity of the desired shape before being solidified. For FGMs preparation there are two kinds of processes classified under graded casting known as slurry dipping and slip casting. Both of these processes are based on a similar principal who says that whenever a porous body is dipped sequentially into slurries with different powder characteristics, the capillary forces will drag the liquid to enter the pores and moves from the surface layers with a stepped gradient behind (Nan, 1993). Some of the processes classified under graded casting process are discussed in the following subtopics.

Centrifugal casting

An investigation on the microstructure and composition gradients in some aluminum based FGMs including Al/SiC, Al/Shirasu, Al/Al_3Ti, Al/Al_3Ni, Al/Al_2Cu combinations have been made by evaluating the dispersion of the different phase particles within the FGM structures fabricated via some different centrifugal casting processes (Watanabe et al., 2005). The study found that Al/SiC, Al/Shirasu and Al/Al_3Ti FGMs can be fabricated by the centrifugal solid-particle method while the different technique of centrifugal *in-situ* method is suitable for the fabrication of Al/Al_3Ni and Al/Al_2Cu FGMs. The combination of both processing methods then are required for Al/(Al_3Ti+Al_3Ni) hybrid FGMs. Although centrifugal casting is a practical mechanism for FGM fabrication as it has the feasibility of scaling up to mass production while maintaining the low cost, this method is only limited for the fabrication of FGM with continuous gradient in the composition.

The phase compositions of FGMs fabricated using this approach are strongly depend on the condition of centrifugal sedimentation process including the duration time, rotation speed, solid and dispersive fluid contents (Jaworska et al., 2006). Self-propagating high-temperature synthesis reaction is added as one of the step followed by the centrifugal casting in the fabrication of TiC-reinforced iron base (Fe-TiC) FGM. The observation of the fabricated specimen indicated an increasing trend of the hardness profile from the outer surface to the TiC-rich inner surface. The wear performance of the TiC-rich inner face was found better compared to the particle-free outer surface of ferritic steel matrices. Centrifugal mixed-powder method (CMPM) shown in Figure 4 is another method introduced as a solution to the limitation of centrifugal casting method in fabricating FGMs containing nano size particles (Gowtam et al., 2008).

The study has been extended to propose another method called reactive centrifugal mixed-powder method (RCMPM) which able to produce better surface properties instead of controllable compositional gradients (El-Hadad et al., 2009).

The formation of gradient solidification is another aspect evaluated in the investigation made on the FGMs fabricated via centrifugation. In this study, SiC, B_4C, SiC-graphite hybrid, primary silicon, Mg_2Si and Al_3Ni reinforced aluminum based FGM were prepared by centrifugal casting. The densities and the size of reinforcements were found as two major roles that influence the formation of graded microstructure (Rajan and Pai, 2009). High density particles such as SiC and Al_3Ni form gradation towards outer periphery while the particle having lower density such as graphite, primary silicon and Mg_2Si form gradation towards inner periphery. The B_4C particle which has closer density to Al alloy is the only particle that distributed more randomly compared to the other systems. Considering the processing of slurry form raw materials, another step named floc-casting has been proposed to be implemented after the centrifugation. During the fabrication, the floc-casting at 80℃ is applied on the SiO_2-Mo FGM to form the homogeneity of the slurry green body before being fired at 1750℃ for 10 min in Ar. Floc casting was found beneficent to control the slurry characteristics and henceforward making the centrifugation of slurry successful.

Slip casting

TZP/SUS304 FGM was developed using slip casting technique (He et al., 2008). The gradual distribution of the chemical composition and microstructure of the fabricated specimens can eliminate the macroscopic FGM interface that occurred in traditional ceramic/metal joint. From this study, the constitutional variation was found influencing the mechanical properties of fabricated TZP/SUS system. Using the same method of slip casting, titanium foams with high purity and high strength macroporous elements were successfully fabricated within addition of emulsion process to stabilize the titanium (Neirinck et al., 2009). During the process, the stability of the emulsion activity is increased by partially hydrophobized the titanium particles. This is important as the emulsion properties is the parameter that influencing the porous properties of the resulting materials. As density is another important consideration to evaluate the emulsion properties, material that can reduce the sintering temperature and the time needed for high density struts should be selected as the struts element.

For this purpose, titanium hydride has been used as the struts in the study. The final product of titanium foams were found having compressive strengths ranging from 120 to 150 MPa. Al_2O_3/W (tungsten) FGM which has potential in the application as a conducting and sealing component for high-intensity discharge lamps (HiDLs) is another FGM that was successfully fabricated via slip casting method (Katayama et al., 2011).

The oxidizing properties of Al_2O_3/W FGM specimens made from two types of W powders have been evaluated for the discussion. In the process, the oxidized W was prepared by heat treatment at 200℃ for 180 min in air before mixed together with the alumina powders in ultrapure water by ultrasonic stirring. The slurry was then cast under controlled pressure into the mold which had a base of porous alumina shape. The study found that the larger ζ-potential of the oxide layer coated on the *W* powder core induce the dispersion of the oxidized *W* powder.

Tape casting

Tape casting has been widely applied in the fabrication of FGMs for various applications. In tape casting process, a powder is blended into an organic solvent with suitable binders and plasticizers to form a slurry with a viscosity ranged from 500 to 6000 mPa·s. The slurry is then cast into a film shape to form a tape which is in size of several µm to mm thickness. The individual layer processing is completed by drying process to remove the residual solvent trapped in the tape. Laminating process under elevated temperature ranged from 50 to 200℃ and pressure of 3 to 30 MPa will follows for the construction of the other layers. The final processing of the laminate involves heating at elevated temperature to remove the organic binder and hot pressing the film to full density. Mg-Cu FGM system were cast into a series of tapes with continuous composition change ranging from 100% Mg to 100% Cu for the use as light-gas gun impactors (Martin and Nguyen, 2005).

Fabrication of graded impactors using this technique has shown significant interest for providing improved control of the pressure profile in gas gun experiments. For a deeper exploration on tape casting process of composites, the calculation of the FGM tape thickness results were characterized by wide adaptability, the blade gap, the casting speed and considering the slurry rheological property into the calculation (Liu et al., 2012). The glass-transition temperature (T_g) and modulus of elasticity (E) are the other two aspects evaluated from the fabricated via tape laminating process of epoxy (EP)/ polyurethane (PU) FGM system. These two materials of EP and PU have E=3.2 GPa, T_g=162 ^{0}C and E=0.069 GPa, T_g=-54 ^{0}C, respectively. The finite element simulations under a steady-state and non-uniform temperature field made on the FGM structure show that the temperature and thermal stress distribution were decreased along the graded direction (Liu et al., 2004).

Thermal spraying

Thermal spraying is a technique by which a structure is coated with melted materials through spraying process. Relating the process with FGMs preparation, the melted materials will be the functionally varied materials which built the coating layers. In this process, the coating precursor is heated either electrically or chemically. One of the advantage of using this technique is the coating can provide thick coatings (20 µm to mm) over a large area at high deposition rate better than the other coating process such as electroplating and vapor deposition. In 2002, three types of functionally graded thermal barrier coatings (TBCs) as well as duplex coatings with the same thermal resistance have been designed in order to investigate the thermal fracture behavior of FGM structures.

In the study, partially stabilized zirconia (PSZ)/IN100 FGM shaving mixed microstructures and PSZ/Inco718 having coarse microstructures were fabricated using slurry dipping and hot isostatic sintering methods to be the specimens. The study found that the specimens fabricated via duplex coatings method could have better or improved spallation life under cyclic thermal loads (Kawasaki and Watanabe, 2002). An approach called shot-control method which applied the concept of gun spray process is another method for functionally graded TBCs fabrication. Using this method, the $NiCrAlY/ZrO_2$ FGM was prepared by spraying technique which done in the form of multi layered coatings with a compositional gradient along the thickness direction. Finely mixed microstructure of metal and ceramic phases and no obvious interfaces were observed at the interfaces of the resulting materials (Kim et al., 2003).

The process was considered reliable when the individual properties of the composed materials in the fabricated FGM were maintained without any phase transformation found. Another FGM that successfully fabricated via thermal spraying process is the FGM that composed by TiO_2 membrane and planar/tubular porous metallic material. In the fabrication, it was found that the intermediate layers made of the same materials are necessary for the prevention of the penetration of the powder suspension into the pores of the support. The ultrasonic wave and grinding methods were found beneficial for the imperfection minimization for the structures (Zhao et al., 2004). Spark plasma technique under thermal spraying process in another study was used for the fabrication of FGM composed by Hydroxyapatite (HAp) and titanium nitride (TiN). The FGM was prepared by sintering at range 1100 and 1200°C under pressure of 150 MPa. The resulting material has Brinel hardness of HB60 and this value was uniformly distributed at the whole composition (Kondo et al., 2004).

In order to improve the adhesion between the adjacent graded layers of FGM, a proper bond coat should be introduced. It is believed that by arranging smooth change on the thermal expansion coefficient mismatch of the composition, the delamination within the FGM structure could be handled. The investigation made on HAp/TiO_2 FGM fabricated via plasma spraying technique found that the crystallinity of HAp and the Vicker's hardness are increasing with the raised temperature. However, at high temperature more than 750°C, the stress induced by the re-crystallization promoted the propagation of cracks and failure on the interface. In another study, the influence of powder characteristics on the mechanical properties of air plasma-sprayed Yttria-stabilized zirconia (YSZ)/mullite coats deposited on SiC substrates have been investigated using indentation testing setup with loads between 10 and 500 mN. The as-spraying coatings were performed at the temperature of 1300°C for 500 h in water environment. The study found that elastic modulus and hardness properties of the resulting materials were highly depend on the size distribution of the starting powders (Cojocaru et al., 2010). Using the same approach of spark plasma sintering (SPS), tungsten carbide/cobalt (WC/Co) FGM was also successfully fabricated (Eriksson et al., 2012).

Laser cladding

Two or more dissimilar materials are bonded together using laser intercession in laser cladding process. During the process, the material which is in powdered form is injected into the system which is built certainly for cladding process while laser which causes melting to occur is deposited onto the substrate. At this time, there will be an interaction between the injected powder stream and the laser. The track of solid material will be produced when the material solidify while the substrate is moved away from the laser spraying area. The involvement of high technology system is there in the laser cladding process when the movement of the substrate and the other parameters need to controlled by computer aided design (CAD) system.

Although the technique has becomes the best technique for coating various shapes and declared as the most suitable process for graded material application, the limitation still exist when that the process needs high costing for the high technology system setup and it is unsuitable for mass production due to the layer by layer process. The appropriate processing via selective laser sintering (SLS) technique which based on free-form concept parameters for the fabrication of polyamide 12 and multi-walled carbon nanotubes (MWCNTs) have been investigated (Paggi et al., 2012). The laser energy density was adjusted to improve the properties of the resulted material properties such as density, flexural modulus and stress distribution at 10% elongation. The study found that the overwhelm mixing of the MWCNTs content compared to the polyamide content interferes the laser sintering process. Compositional gradient distribution and bonding strength of FGM that fabricated

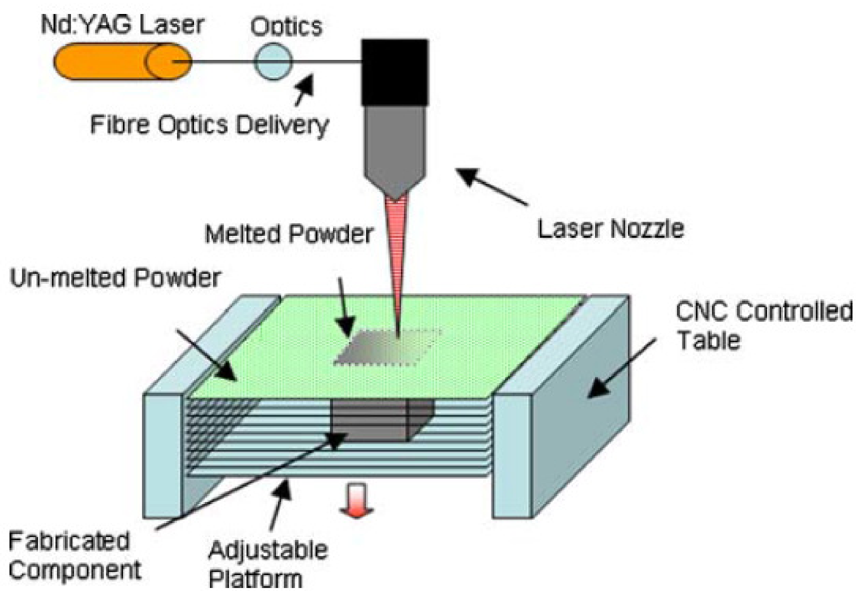

Figure 5. Experimental setup for laser assisted processing using Nd:YAG laser power source (Mumtaz and Hopkinson, 2007).

based on rapid prototyping within laser cladding technique are the other two properties evaluated in a different study. The final WC-NiSiB alloy FGM products which possess the improved highlighted properties fabricated in this study was found applicable for the use in high-temperature tribological applications. The study mentioned that the surface roughness and the geometrical properties of the synthesized FGMs can be controlled by adjusting the heat input during the laser cladding process (Ouyang et al., 2002).

The effect of different laser strategy is another aspect investigated to improve the laser cladding process in producing FGMs. In order to see the correlation between the laser strategy and the FGM properties, the dispersion of laser fusion of different weight proportion of Cu (0, 25, and 50%) powder in H13 was analyzed. From the analysis, refill strategy was found as the best technique as it produced the lowest porosity of H13 with fine dendrite structures (Beal et al., 2006). Characterizing the properties of the fabricated FGM is another issue that should be highlighted in the study of this field. The theoretical principles used in photo thermal displacement (thermal mirror) method which is constructed for measuring the physical properties of the opaque multilayered and graded coatings with low thermal conductivity was analyzed (Elperin and Rudin, 2007).

This method proved that the properties of the laser assisted coatings including the thermal diffusivity and the coefficient of linear thermal expansion are feasible to be determined as the two-dimensional thermal elasticity, the expressions for the photo induced displacement, the slope of the coating surface as a function of time and the physical properties of the sample were analytically solved. The suitability of using FGMs in the applications subjected to thermal fatigue is another aspect investigated in a different study. During the FGM specimen fabrication, two different filler materials from AISI H13 tool steel have been used and the content of the silicon were varied to make the gradation of the secondary carbide content. The study found that the thermal fatigue resistance was decreasing with the increasing of the silicon content in the filler materials (Fazarinc et al., 2011).

In 2005, the pulse-periodic Nd:YAG shown in Figure 5 with wavelength of 1.06 µm was found as the more preferable laser source for the development of FGM components. This technique allows the development of pre-defined compositional gradients which is done by changing the powder composition to be in appropriate composition values. The resulted FGM fabricated via this technique shows minimal transition zone of only 70 µm between the composed materials (Yakovlev et al., 2005).

The Nd:YAG laser power type was also being used in a fabrication via selective laser melting (SLM) of super nickel alloy and zirconia FGM. The resulting materials contained an average porosity of 0.34% with a gradual change between layers without any major interface defects (Mumtaz and Hopkinson, 2007). Solid freeform fabrication (SFF) and laser-based flexible fabrication (LBFF) methods which developed based on laser cladding principal are the other two methods introduced for fabrication of FGMs. By using these methods, a hollow square mold insert with additive layers of H13 steel. Ni/Cr alloy and TiC was successfully fabricated using circular and rectangular beam (RB) profiles determined using ANSYS analysis. The fabricated FGM mold insert has excellent integrity, beneficial microstructure, strong interfaces and high hardness.

These good properties were confirmed through the comparison with the H13 steel molds which tested together in a thermal fatigue environment that performed to evaluate the ability of the structures in resisting the crack initiation, thermal strain and oxidation (Jiang et al., 2005). Laser rapid manufacturing is another method introduced to be under laser assisted processing. Inspiring this method, a bi-metallic tube (cobalt based alloy Stellite 21) and austenitic stainless steel (316L) was fabricated using a 3.5 kW CO_2 laser which is integrated with a beam delivery system, a 5-axis CNC work station and a power feeding sub-system. The fabricated specimens exhibited the desired transition in chemical composition and hardness across its wall thickness. In order to achieve the proper dimensional control and desired heterogeneity of the chemical composition of the specimens, the study emphasized that it is necessary to monitor the processing parameters accurately using on-line control system (Ganesh et al., 2009).

The on-line control system then could be seen developing when three digitally controlled processes known as 3-D laser cladding, 3-D laser-engineered net shaping (LENS) and selective laser sintering (SLS) were introduced in the heading of rapid prototyping (RM). These three methods are used to obtain coatings of FGM by direct powder injection into the laser beam, from CAD solid models with minimal lead times especially for metal art fabrication and constructing 3-D FGM structures through the layered manufacturing, respectively. The main advantage of all these methods is focused on the short time interval needed for processing completion (Joshi et al., 2012).

Vapor deposition method

Vapor deposition is a process by which materials in a vapor phase are condensed to form a solid material. This process generally is being performed to make coatings for the alteration of the properties of the substrates such as in term of mechanical, electrical, thermal, wear and etc. Basically, vapor deposition is classified into two categories namely chemical vapor deposition (CVD) and physical vapor deposition (PVD). In order to produce the desired deposit using CVD process, the substrate is exposed to the volatile precursors to allow the reaction and decomposition on the substrate surface. The chemical reaction part in CVD coating is replaced with purely physical processes such as high temperature vacuum evaporation or plasma sputter bombardment in PVD coating. The processing sources distinguish the two different methods under vapor deposition process.

C-based materials which has an excessive chemical sputtering which yield at 600 to 1000 K and exhibits irradiation which enhanced sublimation at >1200 K when exposed to plasma erosion condition were successfully fabricated via CVD method in 2002. The problem of serious C-contamination of plasma was solved by chemically deposited the SiC coatings on the surface of C-substrate. Although the C-based materials such as SiC/C, B4C/Cu SiC/Cu and B4C/C bulk FGM were successfully fabricated using this method, the performance of the resulting materials were considered not satisfying when there were some crack initiation which believed causing by the heating and cooling cycles applied during the fabrication observed on the structures (Ge et al., 2002). One way to overcome the crack initiation is to reduce and manage the relaxation in the thermal stresses on the structures. Low pressure chemical vapor deposition (LPCVD) method was applied in the fabrication of FGM consisting of carbon fiber reinforced carbon (C-C) and SiC coating layers in order to see the rate of thermal stresses reduction (Kim et al., 2003). During the fabrication, after designing the compositional distribution of the SiC/C FGM interlayer, the deposition conditions of the entire compositional range of the structures were determined using thermo-dynamic calculation to reveal the thermal stresses.

The experimental results proposed the SiC-rich compositional profile in the FGM interlayer as the most effective profile for relieving the thermal stress (Kim et al., 2005). Different deposition method called diamond like carbon (DLC) based is another method introduced under vapor deposition method. The tendency of the DLC coated FGM to peel off because of the high contact stresses and deposition thickness has becomes the main limitation of using these materials. The mechanical, microstructure and tribological behavior of two films (Ti-a:C and Ti-TiC-a:C) that deposited on AISI M2 steel using closed field unbalanced magnetron sputtering (CFUBMS) method were investigated. The study concluded that the incorporation of a TiC buffer layer between the Ti interlayer and the a:C matrix contributes to the improvement in the hardness, wear rate and adhesion properties of the resulting materials (Bulbul and Efeoglu, 2010).

For the manufacturing of both an oxidation resistance SiC coating and functional SiC film using CVD process, CH_3SiCl_3 or methyltrichlorosilane (MTS) is needed as the most important precursors. The decomposition product of MTS was investigated using thermodynamic calculation after obtaining the correct thermodynamic data from the authoritative data sources. The G3(MP2) has been applied to evaluate the thermodynamic data of MTS (gas). The analysis has been conducted based on theories of G3, G3//B3LYP and G3(MP2)//B3LYP and from variables scale factors for G3(MP2). The final fitted equation for MTS (gas) found was $\Delta_f G_m^0 = 7.5763 \times 10^{-6} T^2 + 1.9649 \times 10^{-1} T - 5.4817 \times 10^2$ where T is the absolute temperature (Zeng et al., 2006). The same material of SiC was used to fabricate Ni/SiC FGM but using different method of eletro-deposition. The observation on the fabricated FGM found that in the direction of the deposit growth, the content of the SiC powder increased gradually while the crystal size decreased until 27.5% (Wang et al., 2004). The process of sub-monolayer formation of mono-sized silica particles which have a remarkable similarity with the atomic thin-film growth process of molecular beam epitaxial (MBE) process have been discussed as a function of deposition time during electrophoretic deposition (EPD) (Sarkar, 2004). The study highlighted EPD process as a powerful and versatile forming and consolidation technique as it can produce homogeneous and dense green bodies with complicated shapes and allow flexibility in microstructural manipulation.

CONCLUSION

This paper presents a review on various techniques applied for fabrication of FGMs. The techniques include PM methods, infiltration process, graded casting processes, thermal spraying, laser assisted process and vapor deposition methods. It can be concluded that the processing techniques of FGMs has reached a considerable level of maturity. It is clear that every technique have its own advantages and disadvantages from different engineering applications point of view.

Some aspects were highlighted as the challenges in developing innovative technique for FGMs preparation such as the adaptability for mass production and up-scaling, the process repeatability and reliability, the cost-effectiveness and the convenience level in controlling the quality. Based on these criteria, this study concluded that the powder metallurgy as the most suitable method for the manufacturing of FGMs in the future works. It is believed that the main issue in implementing the PM method which is the sintering process should be further explored in order to achieve improvement in the microstructure and mechanical properties of the resulting FGMs.

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