# Concept and P/M Fabrication of Functionally Gradient Materials

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Abstract: Graded structures may be found in ordinary engineering materials from former days; however, if one has begun to tailor the intentional gradient of composition and/or microstructure in a material in order to achieve the desired functions and properties, the material is known as a Functionally Gradient Material (FGM). The FGM has great potential for applications in many fields by using gradient on chemical, biochemical, physical and mechanical properties. This paper briefly describes the concept of FGM, with special reference to the large progress of research works on thermal barrier materials where the role of thermal stress relaxation function has been emphasized. Powder metallurgical processing of this type of FGM is reviewed on the basis of recent activities. © 1996 Elsevier Science Limited and Techna S.r.l.

#### 1 INTRODUCTION

Functionally Gradient Material (FGM) is a novel concept for the realization of innovative properties and/or functions that can not be achieved by conventional homogeneous materials.<sup>1,2</sup> In its simplest structure, it consists of one material on one side, a second material on the other, and an intermediate layer whose structure, composition and morphology vary smoothly from one material to the other at the micron level (Fig. 1).<sup>3</sup> Their transition profiles must be pre-designed and introduced intentionally in order to achieve the desired function. This means that FGM may undoubtedly be classified into a distinct category separated from conventional homogeneous composites and nano-composite materials (Fig. 2).<sup>4</sup>

From the historical point of view, the description of the idea of gradient material is found in the literature. However, work was not begun along this line until later. In 1987, a large national project commenced in Japan, entitled Research on the Basic Technology for the Development of Functionally Gradient Material for Relaxation of Thermal Stress, which was aimed at developing superheat-resistant materials for the propulsion system and air-frame of the space plane. Intensive research efforts have been devoted to the project. As a result, the concept

of FGM was virtually shown and the term FGM (Functionally Gradient Material) was introduced for the first time. The first paper on FGM fabrication was reported in 1987.6 The publication of papers concerning FGM has been growing extensively after the two International Symposia on FGM (held in Sendai in 1990 and San Francisco in 1992). There have also been small research projects on composites with similar functions carried out individually by investigators from around the world. The Workshop of Structural and Functional Gradient Materials was held in Germany in 1993, and the Workshop of Functionally Graded Structural Materials was held in the United States in March 1994. At present, internationl symposia on FGM are being held on a regular basis throughout the world. The term "FGM" for this unique composite material is now accepted widely in the world. It is evident that FGM holds the key for many applications, which include structural materials, that can endure high temperature exposure with wear or corrosion resistance. Also, they have considerable potential to be extended from structural to electrical, <sup>7</sup> chemical, optical, nuclear and even biomedical areas. Over the past few years, therefore, FGM has received increasing interest on a worldwide scale.

Simple and/or natural "gradient" may arise in ordinary materials. For example, the surface of a

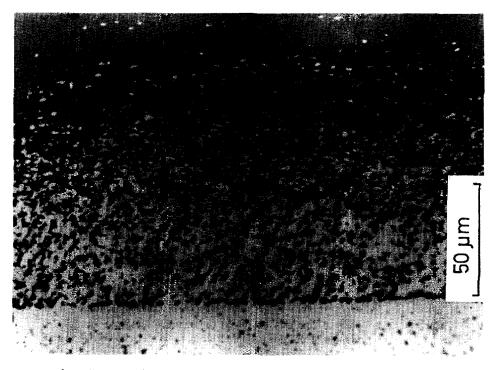


Fig. 1. Microstructure of powder spray formed FGM. Spray formed from alcohol suspension of PSZ and SS304 powders.

|  | Homogeneous<br>material | Functionally gradient Material |
|--|-------------------------|--------------------------------|
| Function/ Property  a) heat resistance b) fracture toughness       | <u>a</u>                | a                              |
| Structure/<br>Texture<br>c) Ceramics<br>d) Metal<br>e) Pore, Fiber | C                       | C e                            |

Fig. 2. Characteristics of functionally gradient material.

nitrided steel, the radial distribution of reflection in glass fibre and the metallic surface modified by ion-implantation. On the other hand, the sophisticated properties, which arise in materials in nature, such as shell, rat incisor<sup>8</sup> and bamboo,<sup>9</sup> are traced to inhomogeneity of chemical composition, structure and morphology in the components of these materials. The fact of this derivation had long been known; however, no attempt to make practical use of it had been made until the national project in Japan. Much of the reason might be that the renovation of homogeneity has played the leading role in the development of advanced materials for years. The Japanese project has shown the possibility of fabricating FGM, yet with the emphasis on

the necessity of a special approach for tailoring FGM, as mentioned later in detail.

From the context mentioned above, this report first describes briefly the concept of FGM. Then, the approach for tailoring FGM is shown, with reference to the thermal stress relief type of FGM. Finally, fabrication of FGMs by the relevant method, particularly focusing on the powder metallurgical process, will be demonstrated.

# 2 CONCEPT OF FGM AND ITS CREATING STRATEGY

The concept of "Functionally Gradient Material" that is applied in general, clearly refers to the realization of innovative properties followed by inhomogeneity that can not be performed by conventional homogeneous materials, emphasizing the following two essential features. The first essential feature includes the tailoring of chemical composition and microstructure in an intentional artificial manner on the basis of the quantitative prediction of the profile of properties distribution to achieve the desired function. The second essential feature includes the availability of fabricating processes which have good reproducibility, as an advanced technology for gradient. The processes must have the capability of producing a precisely controlled profile of chemical composition and must be directly fabricable from the results of computation performed by the material designers. Having achieved the above essential features,

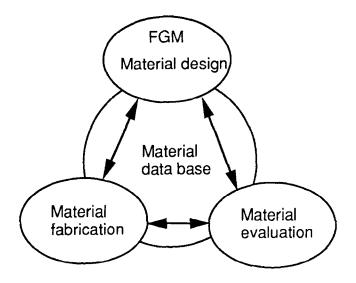


Fig. 3. Approach for FGM development.

namely the integration of gradient function designing and subsequent realization of pre-designed gradient structure, we may really say that a functional gradient has been introduced intentionally into structural or functional materials: that is, functionally gradient materials have been created.

The question is what approach should we take in order to create an FGM that satisfies the essential features. One answer may be found in the approach called materialization of systems, which has been demonstrated for the development of the thermal stress relief type of FGM. As shown in Fig. 3, it consists of a loop of three processes, material design, fabrication and evaluation, linked closely together. The iteration of the loop enables the FGM to become more sophisticated. Although investigations can be focused on each one of the processes of the loop, it is emphasized that the practical fabrication of an FGM must be carried out through the whole loop.

In material design, the compositional profile is optimized to meet the requirement of desired functions by the use of computerized systems,4 in which material properties for different chemical compositions, such as Young's modulus, thermal expansion coefficient, thermal conductivity and Poisson's ratio, are estimated with theoretical methods for preliminary design, or directly referred to the raw experimental values. It is to be noted that there is still no single equation for quantitative property estimation which covers the full range of material composition, taking into account the effect of actual microstructure on properties. Thus, the relation between material properties and chemical composition must be determined by practical experiment with a combination of quantitative microstructural analysis (size, shape, orientation, contiguity, pore, etc.). To perform the optimization

Table 1. Fabrication processes of FGM

| Phase  | Process           | Materials combination                               |
|--|-------------------|---|
| Gas  | CVD               | SiC/C, SiC/TiC, TiC/CC/C-C composite, C/ceramics    |
|  | lon plating       | TiN/Ti, TiC/Ti, ZrO <sub>2</sub> /Cu, C/Cr          |
|  | Plasma spraying   | YSZ/NiCrAIY, YSZ/NiCr,                              |
|  |                   | diamond/WC, YSZ/Cu                                  |
| Liquid Electrodeposition<br>Plasma spraying<br>Eutectic reaction | Electrodeposition | Ni/Cu   |
|  | Plasma spraying   | YSZ/NiCrAIY, YSZ/Ni-Cr                              |
|  | Eutectic reaction | Si/ZrSi <sub>2</sub>                                |
| Solid SHS  Powder metallu  Diffusion                             | SHS               | TiB <sub>2</sub> /Cu, TiB <sub>2</sub> /Ni, TiC/Ni, |
|  |                   | $MoSi_2$ -SiC/TiAl, $ZrO_2/Ni$ ,                    |
|  |                   | PZT/Ni, PZT/Nb                                      |
|  | Powder metallurgy | YSZ/SS304, YSZ/Mo,                                  |
|  |                   | YSZ/Nb, SiC/AlN/Mo,                                 |
|  | Diffusion         | Si <sub>3</sub> N <sub>4</sub> /Ni, W/Cu, Ni/Al     |

of the compositional profile, we have to determine the design criteria on the basis of the desired function. In the case of the thermal stress relief type of FGM, though fracture strength of non-FGM composites was used as a preliminary design criteria, the design criteria was modified so it was based on the fracture mechanism, which contributes to the FGM's failure under actual operating conditions. With the feedback of the design criteria to the materials design, it has been shown that a reasonable design of compositional profile can be achieved.

In the synthesis, FGM is fabricated by using a variety of fabricating processes according to the pre-designed gradient structure. Fabricating processes are roughly classified into three processes: gaseous, solid and liquid processes, which are summarized in Table 1, with examples of application of materials combinations.<sup>4</sup> Gaseous processes allow to prepare directly thin films or plates while controlling gas flow rates and/or temperature.4 The powder metallurgical process is one of the most viable routes for FGM, in which a wide range of composition control and microstructure variation are allowed, along with a shape forming freedom.<sup>2</sup> Also, this process allows to obtain a bulk material, as well as thin coating layers.<sup>3</sup> The self propagating high temperature synthesis (SHS) is characterized by easy sintering of the bulk material in a relatively short time period.4

The FGM is evaluated by using relevant performance tests and the results are fed back to the design process and stored in the database. The performance tests include the local evaluation of microstructure and material properties to show the performance of designed structure and properties distribution. They also include the evaluation of overall performance of FGM properties. Although

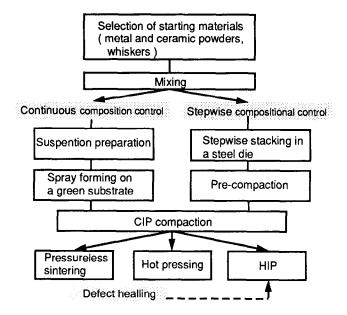


Fig. 4. A flow chart of P/M fabrication of functionally gradient material.

conventional methods may be applicable, some modifications, or the development of new methods, are necessary because of difficulties in measurement due to the continuous change of properties in the local region. Moreover, fracture behaviour in a ceramic/metal FGM may change from brittle to ductile fracture due to the gradual variation of the contiguity of ductile, metal phase. The overall mechanical behaviour of FGM has to be evaluated, not only on the macroscopic scale, but also on a microscopic scale, such as damage growth, microcrack initiation at interfaces and crack propagation. In many applications, FGMs may be exposed to constant or alternating thermal loading with high temperature gradient from one side to the other. Therefore, the thermal stability of the FGM structure must be evaluated, because of microstructural change caused by Ostwald ripening and because of unstable compositional distribution due to the diffusion of structural elements. Also, thermal shock resistance, thermal fatigue characteristics, temperature profile and overall heat flow must be evaluated. The establishment of a performance test is becoming of extreme importance, because it enables us to evaluate the secured functions and the structural integrity in the prepared gradient materials in order to use FGMs in a practical way. Although the performance test for FGMs has to be urgently developed, only a few methods are available at the moment, which are found in some scientific reports addressed on this topic. 11-14 The final validation of fabricated FGMs is manifested through the performance test; great effort must be focused on the establishment of a standardized performance test.

#### 3 P/M FABRICATION PROCESS OF FGM

The powder metallurgical process is a very viable and suitable route for FGM fabrication.<sup>2,15</sup> It includes the following sequential steps (Fig. 4): the selection of a material combination of metals and ceramics; design of the optimum compositional distribution; step-wise or continuous stacking of powder premixes according to the pre-designed compositional profile; die compaction and subsequent cold isostatic pressing of the stacked powder and sintering of the pressed compact. The key points are essentially two-fold: how to obtain the graded green compact and how to sinter the graded green compact at uniform sintering temperature without defect formation. The defects, such as large residual porosity or small cracks in the compacts, are healed, if necessary, by HIP.

Concerning the first point, several methods have been proposed and demonstrated. The simplest one is step-by-step stacking of mixed powders of different compositions. 6,16,17 Other methods which enable to make continuous stacking with changing composition include the wet filtration process, 18 the vibrating stacking process, 19 the centrifugal process,<sup>20</sup> the wet powder spray forming process,<sup>21</sup> sequential slip-casting<sup>22</sup> and the slurry dipping process.<sup>23</sup> These processes sometimes require the use of binders to stabilize the green bodies, necessitating a de-waxing process similar to metal injection moulding. Among them, the powder spray forming process is one of the candidate methods for continuous stacking of mixed powders in a thin layer, as well as for manipulating the three-dimensional gradient structure. Figure 5 shows a schematic diagram of the powder spray stacking system. The apparatus consists of a mixing and supplying system of suspension, a spraying and drying system for deposition and a specimen handling system for 3-D spray stacking. The powder spray stacking process includes the preparation of a suspension of metal and ceramic powders using ethanol as the solvent; adjusting the mixing ratio by controlling the flow rate of the ceramic suspension; leading the mixed suspension to the spray nozzle and subsequent spraying by compressed air; depositing on the pre-heated substrate and drying. The by-passed powder was trapped by a cyclone.

The second problem follows from different sintering behaviour, which occurs in powder compacts with different mixing ratios of metal and ceramics. The sintering imbalance will cause various faults in the sintered products, such as warping, frustum formation, splitting and cracking.<sup>2</sup> The fine adjustment of sintering behaviours can be accomplished by size control of the starting

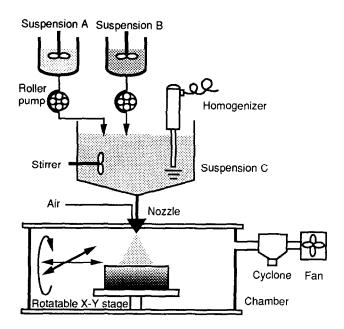


Fig. 5. Schematic diagram of powder stacking apparatus.

powders<sup>2,16</sup> and by the addition of an activating element.<sup>24</sup> The troublesome effect of the sintering imbalance can be largely avoided by pressure sintering, i.e. by hot pressing and hot isostatic pressing. In the case of HIP, the FGM green compacts with unequal density distribution result in a shape fault. The fine adjustment of compacting characteristics by the size control of the raw material powders and the mixing conditions is effective in avoiding this problem.

At present, disc-shaped samples are fabricated by pressure sintering,<sup>25–7</sup> while pressureless sintering is applied to fabricate cylindrical samples.<sup>6,17,24</sup> The minimum dimensional size of composition control depends on the particle sizes of the stacking powders and on the method of powder stacking. The layer-by-layer stacking enables us to control the composition profile with a dimensional size fineness of 0·2 mm. While, by spray deposition technique, the minimum control size of 0·01 mm has been realized.<sup>21</sup> Laser-beam sintering has been developed to utilize the versatility of the P/M process by employing various material combinations.<sup>28,29</sup>

# 4 MICROSTRUCTURE CHARACTERISTIC AND CONTROL OF COMPOSITION— PROPERTY RELATIONSHIP IN P/M FGM

The metal/ceramic functionally gradient material fabricated by the P/M process indicates characteristic microstructural transition with composition, 2,15,30-2 as shown in Fig. 6. The microstructure is characterized by the gradual replacement of the matrix from metal to ceramic, with an increase in the

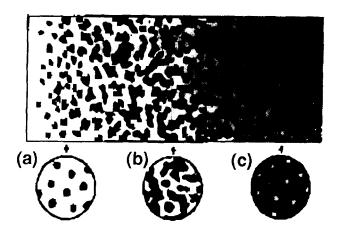


Fig. 6. Schematic illustration of the microstructural transition in a P/M FGM. (a) A dispersive structure, (b) a network structure, and (c) an alternative dispersive structure.

fraction of ceramic phase. In the metal-rich side, ceramic particles are dispersed in the metal matrix. With the increase of ceramic phase, clusters of ceramic are formed and the consequence of its further growth results in the formation of a network structure of ceramic phase. Then, the network of metal phase is gradually diminished and turns into isolated phases dispersed in the ceramic matrix in the ceramic-rich side. Since the network structure may be determined by the formation of percolation clusters of ceramic or metal phase, there is a composition range in which both phases are in a network structure. There is no single quantitative method which is capable of characterizing these microstructures (covering the full range of compositions) by itself. Therefore, it is necessary to characterize the microstructure with multiple methods, such as stereology. topology and fractal methods. The quantitative microstructural analysis by stereology is effective for determining the composition (volume fraction of metal and ceramic), size, orientation and its distribution, while the topological parameter (Betti number)31 is the best for evaluation of the microstructural continuity and percolation composition. Recently the use of fractal dimensions have been demonstrated to present the complexity of the microstructure.<sup>31</sup> All these approaches are of extreme importance for deriving the relationship between composition and material properties.

Physical properties of metal/ceramic sintered composites, such as thermal expansion coefficient, thermal conductivity and electrical conductivity, have a specific compositional dependency. The sinuous curves in these diagrams are explained qualitatively as a transition between two curves which are given by the Maxwell relation for two kinds of dispersion structure (Fig. 7).<sup>2</sup> The compositional dependency on Young's modulus is shown in Fig. 8.<sup>30</sup> The relation is close to a linear

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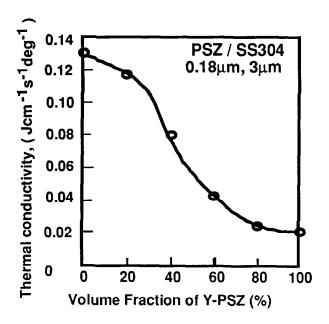


Fig. 7. Relationship between thermal conductivity and composition.

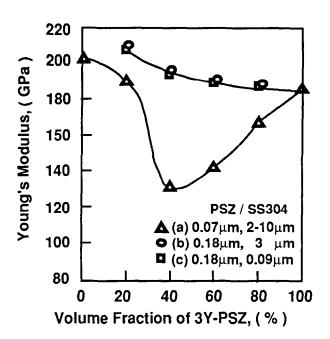


Fig. 8. Relationship between Young's modulus and composition.

relationship, but when examined in detail it exhibits a sinuous curve deviating from the linear relationship. The microstructure, which has rather coarse dispersed phases, is susceptible to delamination at the interface of the matrix. It has been noted that the susceptibility depends on the mismatch of the thermal expansion coefficients and size of the dispersed particles.<sup>34</sup> In this case, property values decrease markedly, as shown with the dashed line in Fig. 8, with extremely large deviation from the linear relation. Quite complicated behaviour in four-point bending strength was

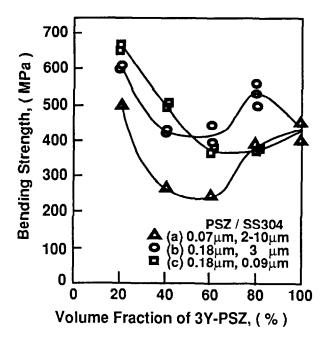
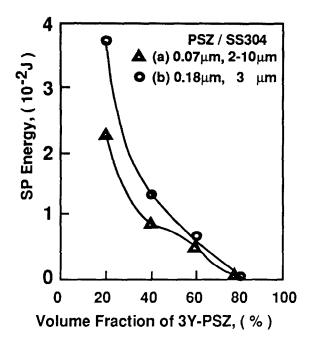


Fig. 9. Relationship between flexural strength and volume fraction of PSZ.

observed in Fig. 9.30 The microscopic fracture mechanism is discussed with AE behaviour, together with the fracture surface observation by SEM.35 It has been demonstrated that the complex behaviour seems to be associated with delamination of the interface, the size and shape of the clusters and formation of a network structure.<sup>33</sup> In FGM where the matrix material turns from metal to ceramic, the transition in fracture mode goes from brittle to ductile. Figure 10 shows the dependency of absorbed fracture energy on composition employed by the small punch test.<sup>33</sup> The transition from brittle to ductile fracture is clearly seen to occur in a wide composition range, with completely brittle behaviour at a PSZ volume fraction of about 80%. The results of fracture surface observation by SEM are shown as a fraction of fracture mode. With respect to the relation between SP energy and composition, the change in fracture mode can also be seen.

As mentioned in the previous section, the thermal stress generated during fabrication shows a maximum at the ceramic-rich side of the surface of the FGM sample, which contributes to crack formation. Thus, to fabricate crack-free FGM it is necessary to improve the fracture strength and toughness of the material in the composition range where maximum thermal stress arises. Pertinent to this, it has been pointed out that fracture strength and toughness in the ceramic-rich side must be improved from the view point of the thermal shock fracture mechanism.<sup>14</sup> It has demonstrated that fine uniform mixing of raw material powders is



**Fig. 10.** Ductile-brittle transition with metal/ceramic mixing ratio (SS304/3Y-PSZ).

very effective in improving the strength and toughness of sintered compacts of metal/ceramic powder mixture. The results of SP test have revealed that fracture toughness depends strongly on the amount of contiguity of the metal phase. Thus, the increase of connectivity of the metal phase is highly desirable and it has been demonstrated that the use of metal fibres is useful for this improvement.<sup>36</sup> Finally, fracture strength and toughness must be optimized for each of the compositions, through microstructural control, in order to improve the thermomechanical performance of FGMs.

# 5 FGMs FABRICATED BY THE P/M PROCESS

# 5.1 Thermal stress relief type of PSZ/SS304 FGM<sup>25</sup>

## 5.1.1 Design

In the design of the thermal stress relief type of FGM, thermal stress calculation must be performed for every thermal condition that the FGM will experience, for subsequent determination of the composition profile to minimize the stresses through the compromising process. The thermal stress calculation for the P/M FGM includes the following two conditions: the fabrication and the actual thermal load condition. For the fabrication, the thermal stress generated during cooling from the sintering temperature for the first, is to be minimized. For the actual condition, the thermal

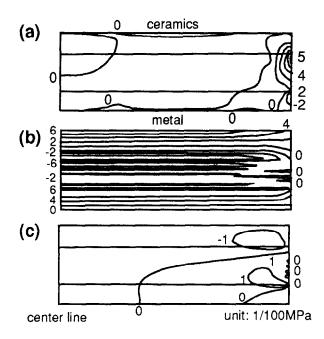


Fig. 11. Contour maps of the thermal stress in the discshaped compositionally graded PSZ/SS304. (a) Axial stress, (b) circumferential stress, and (c) shear stress.

stress generated in a temperature gradient in a thermal barrier situation is to be minimized. There has been no self-consistent design scheme which takes into account both of the thermal load conditions. However, it is to be noted that the optimum composition profile for the stress reduction in the as-sintered FGM is still effective, although not optimum, in reducing the thermal stress concentration in the thermal barrier situation. 15 Figure 11 shows the distribution of axial, circumferential and shear stresses for a disc-shaped sample of partially stabilized zirconia/stainless steel in the case of cooling from 1723 K.<sup>15</sup> The axial stress is largest on the surface of the ceramic-rich region, while the circumferential stress is largest at the centre portion of the top surface of the sample. The maximum values for both the axial and circumferential stresses are about the same. A stress distribution quite similar to that for the circumferential stress has also been obtained for the radial stress. The shear stress is small compared with the axial and circumferential stresses and can be ignored in the present discussion. These stresses are seen to depend on the sample thickness. For a sample thickness of 2 mm or more, which is the usual size of a P/M FGM, the axial stress at the side surface of the disc predominates. 16 Crack formation is, in fact, often observed for the sample without an optimum compositional profile. Figure 12 shows the existence of the optimum composition profile to reduce the axial stresses, which will promote crack formation at the sample surface.

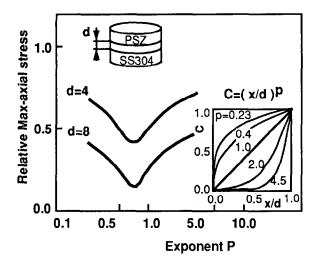
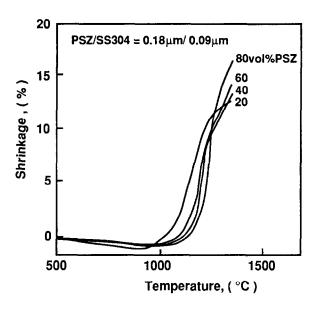


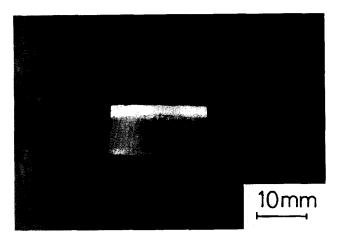
Fig. 12. Maximum axial stress (normalized with respect to direct bonding) as a function of the exponent of the composition profile. x is the distance from the PSZ phase.



**Fig. 13.** Adjustment of sintering shrinkage accomplished by size control of the PSZ and SS304 powders.

# 5.1.2 Fabrication

According to the optimum design of composition profile on the basis of thermal stress analysis, a disc-shaped FGM (30 mm in diameter and 10 mm in height) was fabricated. The thickness of both the PSZ and stainless steel layers is 2 mm and the composition controlled layer is 4 mm. The composition distribution was determined by using the design curves in Fig. 12. In the case of P/M fabrication of FGM, the adjustment of the sintering imbalance in powder compacts with different mixing ratios of metal and ceramics is the first task. Figure 13 shows an example in which the adjustment was performed by size control of raw material powders. Considering the results of shrinkage curves, it is found that the use of PSZ powder of 0.18 µm in diameter



**Fig. 14.** Sintered PSZ/SS304 FGM with an optimum compositional gradient.

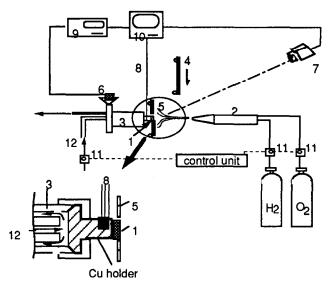


Fig. 15. Burner heating test system and sample setting configuration. (1) Test sample, (2) torch burner, (3) cooling chamber, (4) shutter, (5) protector plate, (6) AE sensor, (7) emission pyrometer, (8) thermocouple, (9) AE apparatus, (10) monitor, (11) regulator valve, and (12) cooling water.

and stainless steel powder of 3 µm is the best combination. The selected powders were mixed and then stacked layer by layer in a steel die: subsequent die compaction at a pressure of 100 MPa and CIP at a pressure of 200 MPa. Sintering was conducted for 1 h at 1623 K under a vacuum below 10<sup>-4</sup> Torr.

Figure 14 shows a macro-view of the sintered FGM compact and its compositional distribution. The shape of the compact is slightly warped due to the difference of shrinkage in each composition, but no defects, such as large residual pores and micro-cracks were observed. The sintered compacts, whose design conditions deviated from the one shown in Fig. 14, caused two kinds of crack: the radial cracks at the PSZ surface due to circumferential stress and the side surface cracks due to axial stress.

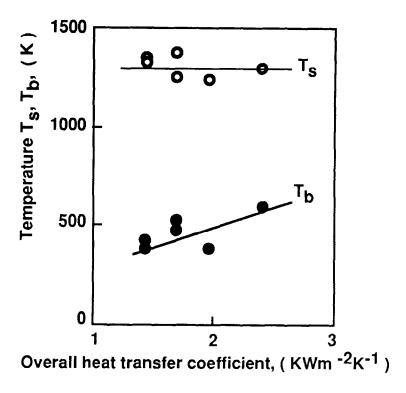


Fig. 16. Critical temperature for crack formation vs thermal resistances of the PSZ/stainless steel FGM.

#### 5.1.3 Evaluation

The differential thermal loading test (burner heating test), as shown in Fig. 15, allows us to evaluate the thermal stress relief function, oxidation behaviour and thermal shock resistance.<sup>14</sup> A temperature difference is given by heating the ceramic side with a burner frame and cooling the metal side with water flow. The damage at the specimen surface is monitored with a microscope and the AE method. In the burner test the generation of large compressive stresses is expected during the heating cycle at the ceramic side of the sample. This compressive stress will cause non-linear deformation at the ceramic surface. As a result, a tensile stress is generated during the cooling cycle at the deformed region, which will cause crack formation. The crack formation due to the resultant tensile stress during the cooling cycle has been actually observed, and the test temperature for the first crack formation can be defined as a thermal barrier performance value for the test sample.

Figure 16 shows an example of the burner test results obtained on disc-shaped FGM samples in the system of partially stabilized zirconia/stainless steel. The test temperature of the first crack formation is plotted against the heat flux estimated with the pertaining temperature differences and is almost constant for the various samples and heating conditions, indicating a material dependency of the thermal barrier property. A study on

the thermal shock fracture mechanism is currently in progress.

# 5.2 Thermal barrier type of SiC/AIN/Mo FGM<sup>27</sup>

#### 5.2.1 Design

The requirements for the top surface of the thermal barrier type of FGM are the retention of strength at elevated temperatures, high temperature of brittle-ductile transition and the heat insulating function. As reported,<sup>27</sup> SiC-50 mol% AlN ceramic alloy has the best heat insulating properties in the SiC/AlN system, which is ten times greater than that of pure SiC. Also, the value of strength at room temperature is maintained up to 1773 K. Therefore, SiC-50 mol% AlN is suitable as a top surface material. On the other hand, Mo was chosen as the metallic material for the bottom surface of the FGM, because the sintering temperature of Mo is close to that of SiC-AlN and it is a heat resistant material. AlN was used for the intermediate layer as a reaction barrier between SiC and Mo. A compositional profile was designed, with a particular emphasis on controlling the heat insulating property of the FGM. The function of thermal stress relaxation during fabrication was not considered in the design, because the thermal expansion coefficients of SiC-AlN, AlN and Mo have almost the same value, and therefore showed

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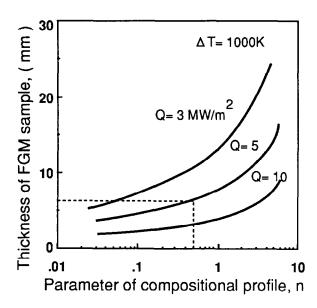
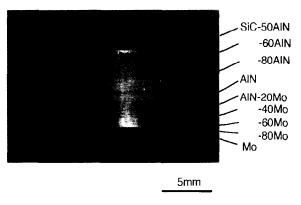


Fig. 17. Design curves for the SiC/AlN/Mo FGM on the thermal conduction of temperature difference of 1000 K.

no significant stress generation during cooling from the sintering temperature. The heat insulating efficiency of FGMs was estimated using the simple theory of heat conduction.<sup>27</sup> The heat insulating property is estimated by the heat resistance, R, which is the ratio of heat flux to temperature difference. Figure 17 shows the design curves for an SiC-AlN/Mo FGM. The exponent of the compositional profile is determined when the thermal resistance and the thickness of the FGM are given for the design condition.

#### 5.2.2 Fabrication

SiC powder with an average particle size of 0.37 µm and AlN powder with an average particle size of 1.47 µm were used as the raw materials. Powder pre-mixes of different mixing ratios were prepared by wet milling for 1-300 h in isopropyl alcohol in a silicon nitride pot with silicon nitride balls. After milling, the wet medium was evaporated. The amount of powder pre-mixes of each composition was weighed according to the predesigned compositional profile of  $\Delta T = 1000 \text{ K}$ ,  $Q = 5 \text{ MW/m}^2$  and t = 7 mm. The powder premixes were stacked layer by layer in a steel die having 16 mm diameter and compacted at a pressure of 100 MPa. The green compacts coated by BN powder were encapsulated in a Vycor glass tube and were HIPed for 2 h at 2123 K at a pressure of 200 MPa. A difference in the compacting characteristics of each powder mixture causes faults such as cracking in the compacts and/or delamination at the interface of step-wise stacked layers during compaction. Also, the green FGM compacts have an unequal density distribution.



HIP: 2123K, 200MPa, 7.2ks

Fig. 18. Macro-view of the HIPed SiC-AlN/Mo FGM sample fabricated successfully.

The fine adjustment of compacting characteristics was accomplished by size control of the raw material powders and the mixing conditions.

Figure 18 shows the macro-view and a cross-section of the HIPed SiC-AlN/Mo FGM. Note that no defects, such as large residual pores or small cracks, were observed in the sintered FGM.

#### 5.2.3 Evaluation

The heat insulating efficiency of FGM for different kinds of material combination has been compared on the basis of the thickness and compositional profile of the FGM. The heat insulating performance, which assumes a heat flux of 5 MW/m² and a temperature difference of 1000 K, can be realized by an SiC-AlN/Mo FGM by controlling the thickness and compositional profile of the graded layer. It is noted that the SiC-AlN/Mo system is a promising material for super-heat resistant FGMs.

## **6 CONCLUDING REMARKS**

The concept of functionally gradient material was briefly described. The FGM is now accepted worldwide, and it has opened new fields for material design and effective applications for industrial materials. The special approach for tailoring FGMs has been shown with reference to the Japanese national project, emphasizing that fabrication of FGMs without material design and the feedback process of material evaluation makes no sense. The powder metallurgical fabrication of FGMs, its pertaining problems and the property-microstructure relationship have been discussed. Finally, the authors agree with the necessity for a worldwide collaboration for effective FGM development, particularly for a material-property database and standardized testing methods.

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