

Fine Bone China Product using Ceramics Slip Rotary Moulding (CSRM) Technique

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ABSTRACT

Bone China is a type of porcelain mainly produced from calcined animal bone. Traditionally, ceramic fine bone china (FBC) products are made by slip casting technique. The traditional technique cannot perform mass production in ceramic industries. Thus, this research focused on improving the productivity of fine bone china product fabrication by introducing a new method called Ceramics Slip Rotary Moulding (CSRM). Basically, the technique of rotary moulding gives high potential for mass production in producing hollow shape green ceramic product. The research compared two formulations of ceramic slip using 100 μ m and 300 μ m FBC powders. The slips were prepared and viscosity for both formulations was measured. Specimens for strength test were prepared and using the CSRM green body, the FBC products were produced and sintered. Laboratory test results showed that slip casting with rotary technique required additives to be added in the slip composition to control and gain sufficient coagulation, fluidity and strength to form a green body. The final slip formulation of 78% FBC solid content and 22% water content for both 100 μ m and 300 μ m powders required 0.2% dispersant (Acumer 9400) and 0.068% coagulant (Duramax B1020). The amount of additives ensured sufficient fluidity for slip to flow smoothly in the mould during the rotation process and form the FBC green body. The formulation was successfully produced for the Ceramic Slip Rotary Moulding (CSRM) and sufficient green strength was achieved to hold the green body during de-moulding and ready for sintering process.

Keywords: *Fine Bone China, Ceramics Slip Rotary Moulding, Hollow Ceramic Product, Slip Composition, Green Body, Sintered Product*

Introduction

As a ceramic material, bone china is a highly specialized product in terms of its appearance mainly due to its translucency, whiteness and high strength [1]. The most common and traditional ways in producing bone china based product is by slip casting method. The bone china body was first developed by Thomas Frye at his factory in 1748, near Bow, London. Cattle bones were transformed into fine bone china with Frye formulae which consisted of 45% cattle bone ash, mixed with other minerals elements that produced finer strong porcelain. However, the term “Bone China” was popularized by Josiah Spode in 1797. Fine bone china as a type of true fine porcelain essentially, was used in tableware and art ware. It features the qualities and notably superior translucency that are associated with bone china. Nowadays, Bone China contains 35-45% bone ash in the clay formula [1-5]. Slip casting method is a common traditional method in ceramic industries whilst slip rotary moulding is an advanced technology in producing hollow ceramic products by rotary moulding technique that requires high solid content and low viscosity introduced in the ceramic industries. It is also known as Integrated Slip Rotary Moulding (ISRM) process [6-8].

Almost all casting slips used in the production of traditional ceramic whitewares consist of plastic ball clay, kaolin, filler and flux in various proportions depending upon application [6]. The ball clay is the most difficult to disperse and is, therefore, normally processed and allowed to age prior to final casting slip preparation. Consistent behaviour of the ball clay and casting slips is vitally important in minimizing day-to-day production problems. Such consistency is essential for the setting of factory production rates which achieve the most efficient and cost effective manufacture of the finished ware [9, 10]. Slip casting method basically requires the use of mould made by plaster of paris. Plaster of paris is a brittle which resemble cement, stone and other porous ceramics, easy to shape and porous solid [11]. The mould will absorb water from clay suspension in order to produce a hollow product at certain wall thickness [9-11]. Slip to be poured into mould is measured within half volume of mould to ensure there is no excess slip produced at the end of the process.

However, sometimes slip requires final adjustments prior to use as their fluid properties change with time [10, 11]. The ageing characteristics of a body will vary depending on its make-up. It is usual to age for at least 24 hours in order to obtain stability prior to slip formulation [11]. This behaviour is the result of the extremely slow rate of reaction between the

various deflocculants and the clay particles in the body system [12]. Because of this slow reaction, slip should be kept for at least 24 hours prior to use in the mould. After storage, the slip was added with coagulant before it can be used in rotational moulding process. Thus, the purpose of this paper was to analyse the composition of Fine Bone China slip formulation and examine the performance of CSRM Process parameters and the properties of green body and sintered FBC product using Ceramic Slip Rotary Moulding technique [13-15]. The advantages of Ceramic Slip Rotary Moulding (CSRM) technique over the traditional slip casting are highlighted in the conclusion session.

Experimental Analysis

Slip Preparation

The cake form of raw fine bone china material was dried 24 hours in electric oven with the temperature of 110°C. It was crushed into small pieces (powder) using mortar and pestles. After that, the fine bone china powder was sieved into two different measurements; 100µm and 300µm respectively. The slip was prepared by mixing together the fine bone china powder and water with the ratio of 78% solid content and 22% water content. Dispersant (Acumer 9400) and coagulant (Duramax B1020) were added into the slip in small volume around 0.2% for dispersant and 0.068% for coagulant that acted as an additive agent. Figure 1 shows the sequences in preparing the slip composition of fine bone china product for Ceramic Slip Rotary Moulding (CSRM) technique.

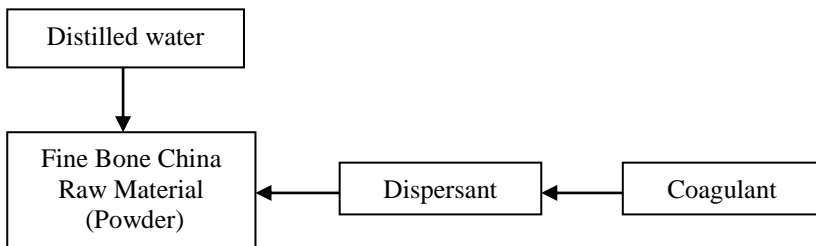


Figure 1: The sequences in preparing the slip composition of fine bone china product for Ceramic Slip Rotary Moulding technique

The slip was then left 24 hours in room condition in order to ensure the composition was stable enough before the next process of Ceramic Slip Rotary Moulding (CSRM). The prepared slip was poured into the egg shape mould made of plaster of paris and underwent rotating process for shaping

purpose. Table 1 shows the chemical analysis of fine bone china, whereas Table 2 shows the chemical analysis of fine bone china obtained using Scanning Electron Microscopic (SEM) instrument.

Table 3 tabulates the composition of polymeric additives (Acumer 9400) which played a key role in the processing of colloidal slip of ceramic. In this work, the dispersion behaviour of slip fine bone china slip and dispersant of polyacrylic acid were investigated by viscosity measurement of the slip. Polymeric additives used were dispersant and binder to help the processing of ceramic bodies. Polymeric dispersant resulted in well dispersed and stable suspensions based on electrosteric stabilization mechanism [17].

Table 1: Analysis of chemical composition of fine bone china material [16]

Element	Weight (%)
SiO ₂	34.04
Al ₂ O ₃	14.92
Fe ₂ O ₃	0.52
TiO ₂	0.10
CaO	26.00
MgO	0.68
Na ₃ O	1.19
K ₂ O	2.00
P ₂ O ₅	19.00

Table 2: Analysis of chemical composition of fine bone china material by SEM process using TM303Plus

Element	Weight %
Oxygen	54.087
Sodium	0.710
Magnesium	0.480
Aluminum	7.622
Silicon	11.448
Phosphorus	8.612
Potassium	0.831
Calcium	16.208

Table 3: Composition of polymeric additives (Acumer 9400) [9]

Component	Concentration
Polycarboxylate	44.0-46.0%
Water	54-56%
Residual monomers	<150.0 PPM

Sintering

The green body product was produced using the slip composition of 78% solid to 22% water content and additional additives such as dispersant and coagulant. After de-moulding, the product was left 24 hours in room condition. The green body product was then sintered in a furnace with the temperature of 1215°C. Figure 2(a) shows the green body product during de-moulding process and Figure 2(b) shows the sintered products. The process parameters for producing green body of hollow shape fine bone china product are as shown in Table 4.



Figure 2: (a) Green Body Product; (b) Sintered Products

Table 4: Process parameters of Ceramic Slip Rotary Moulding (CSRM) in producing green hollow shape fine bone china product for both 100µm and 300µm

Main Parameters	Data input
Rotation Speed, RPM	14
Chamber temperature, °C	90
Rotation time (forming), minute	15
Rotation time (heating), minute	15
Rotation time (cooling), minute	40

Characterization of sintered component

In this experiment, viscosity test was measured by using Gallenkamp Viscometer while strength test was conducted using Universal Testing Machine. For the porosity test, the sintered product was weighted in vacuum weighing instrument to record the weight of the sintered product before it underwent the bath soaking process. In microstructure analysis, the product was performed using scanning electron microscopy (SEM).

Results and Discussion

Slip Formulation

The amount of ceramic prepared was based on the inner size of the rotary mould and thickness required of the hollow product [7, 8, 9]. Before fine bone china slip was poured into the mould, pH levels of the slips were measured right after additives were added to the slip using pH meters. The value of pH level of the fine bone china for both 100 μm and 300 μm was 8.27 respectively, which was alkaline. Based on the first glance, the surface of the green body depended on the quality of the inner surface of the mould [9]. The parting line between male and female mould was formed on the surface of the green body. The observation suggested that the fine bone china slip formulation was suitable to be used in Ceramics Slip Rotating Moulding (CSRM) machine [9, 18]. The viscosity values for both 100 μm and 300 μm are shown in Table 5.

Viscosity

From Table 5, the values of viscosity for fine bone china slip with powder size of 100 μm and 300 μm were 65.0 x 10³ CPS and 67.0 x 10³ CPS respectively. Even though the percentage of solid content for both size of particles was the same, it produced different values of viscosity reading. Therefore, it can be said that the bigger size of particles, the higher the value of viscosity [6, 19, 20]. High value of viscosity will decrease the fluidity of slip [6]. Thus, time for the slip to fully cover the inner surface of mould will also increase. Sometimes, when the fluidity is too low, the slip does not cover the inner mould surface [8].

Table 5: Value of viscosity for fine bone china slip

Slip	Particle Size (μm)	Solid Content (wt %)	Water content (wt %)	Viscosity (CPS)
Fine Bone China	100	78	22	65,000
Fine Bone China	300	78	22	67,000

Strength Test

Table 6 tabulates the average strength test of green body and sintered product for both 100 μm and 300 μm respectively.

Table 6: The average strength test of green body and sintered product for both 100µm and 300µm

Size of FBC (µm)	Force (N)	
	Green Body	Sintered
100	11.82	241.08
300	8.15	175.10

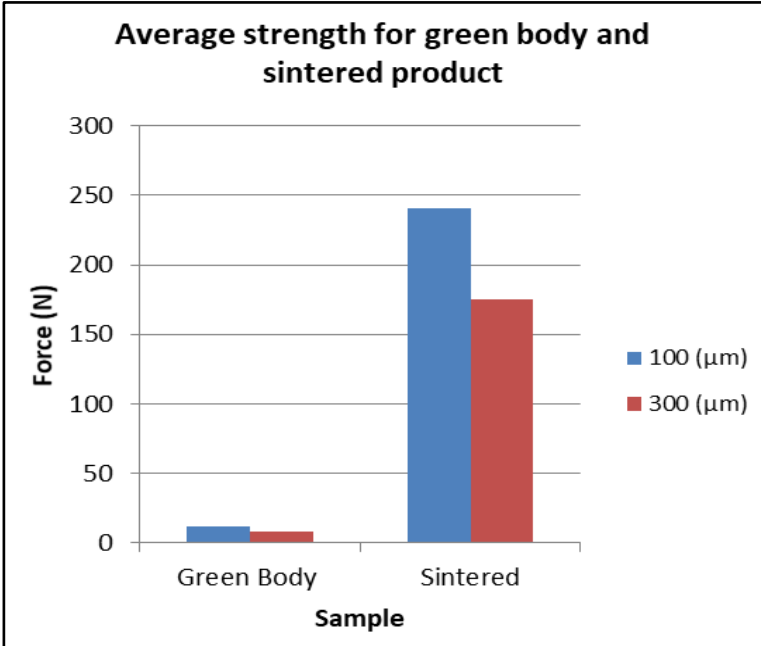


Figure 3: Strength test of green body and sintered product for both 100µm and 300µm

Based on the plotted graph in Figure 3, it can be said that the sintered fine bone china samples for both 100µm and 300µm were 241.1N and 175.1N which gave more strength compared to the green body product where the maximum forces were 11.82N and 8.2N. As the sintered product underwent sintering process at 1215°C, the material properties of the fine bone china became stronger compared to the green body product. The particles in the sintered product bonded together and formed a tough product that was not easily broken. When compared between 100µm and 300µm, the strength of 100µm was higher than the 300µm for both green body and sintered samples. It means that size of particles also plays an important key role in order to produce a dense high strength and toughness of bone china

product [4, 8, 19, 21, 22, 23, 24, 25, 26, 27]. Sintered sample also shows that the smaller size of particle gives high strength to the product.

Porosity Test

Porosity or pore space is the amount of air space or void space between particles. The porosity of particles is the ratio of the volume pore space in a unit of material to the total volume of material. The arrangement or packing of particles plays a role in porosity [19, 26, 28, 29]. The porosity, however, is not related to the particle size but rather to the way particles are packed. The narrower the size range of the aggregate particles, the higher the porosity [26, 30]. Generally, ceramics powder with a narrow range of particle sizes between $45\mu\text{m}$ and $175\mu\text{m}$ are more uniform and suspensions can be produced.

Figure 4 shows the percentage of porosity for both $100\mu\text{m}$ and $300\mu\text{m}$ of sintered product. The value of porosity percentage for $300\mu\text{m}$ was higher than $100\mu\text{m}$ where the values were 26.90% and 20.62% respectively. Thus, the porosity percentage of $300\mu\text{m}$ bone china was much higher than $100\mu\text{m}$. As related to the strength of the sintered product of both $100\mu\text{m}$ and $300\mu\text{m}$, it showed that the strength of product was influenced by the percentage of porosity. The higher the percentage of porosity value, the lower the force that can be exerted by the product [24, 26, 31, 32].

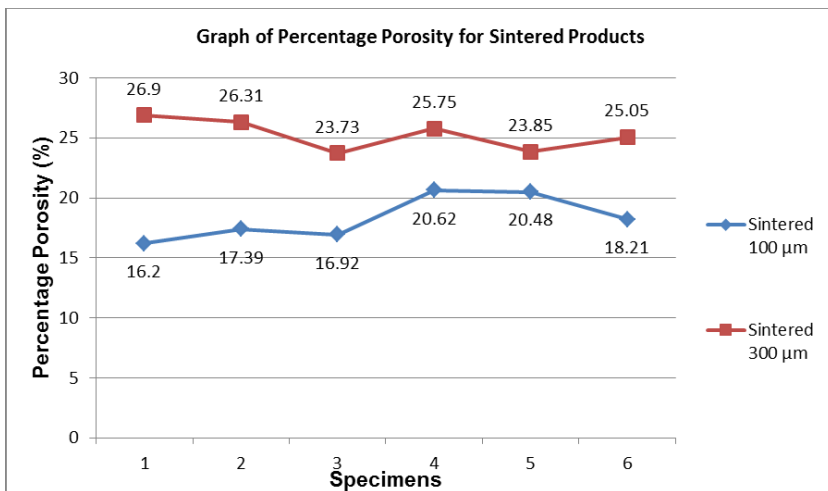


Figure 4: Percentage of Porosity for both $100\mu\text{m}$ and $300\mu\text{m}$ of sintered product

Scanning Electron Microscopic (SEM)

Figure 5 (a) and (b) and Figure 6 (a) and (b) show the SEM analyses using TM303Plus of green body and sintered fine bone china products for both 100 μm and 300 μm respectively. The fracture surface of dried product showed the powder particles agglomerated together as can be seen in Figure 5(a) and (b). After sintering process was completed at 1215 $^{\circ}\text{C}$, the surface of the sintered product showed the powder particles formed together homogeneously; hence, forming a strong ceramic body for producing hollow ceramic product compared to dried product as shown in Figure 6(a) and (b).

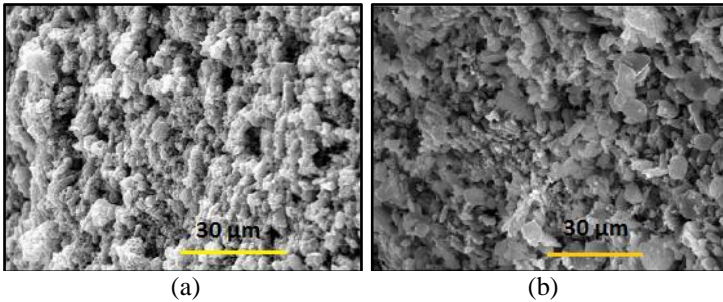


Figure 5: (a) Green Body 100 μm , (b) Green Body 300 μm

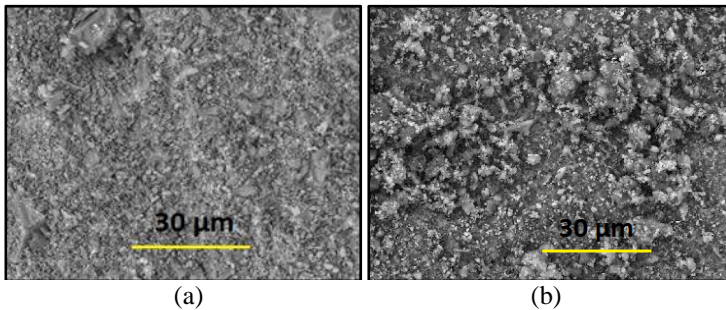


Figure 6: (a) Sintered 100 μm , (b) Sintered 300 μm

Conclusion

The characteristics of raw materials are important in maintaining quality of hollow fine bone china product during the manufacturing process. It is also important to observe that the formulation of the slip with varying factors influences the working fluid volume flow rates. The chemical composition, particle size distribution, and colloidal surface affected the sintered and green body properties of the fine bone china samples. In the suspensions formed,

the properties evaluated were the composition of solid content, pH level and viscosity of the slip. For green body, the properties evaluated included viscosity, fluidity, and strength. On the other hand, strength, porosity and shrinkage were measured for sintered fine bone china. Based on this research, it showed that a formulation of fine bone china with solid content of 78% for CSRM technique was successful. Due to the results gained from the viscosity, strength and porosity tests of the product specimens made from the compositions, it was able to form a good high strength quality to dense ratios product within a short period of time. Therefore, ceramic slip rotary moulding (CSRM) technique results consistency of wall thickness, and a variety of complex shapes can be produced with no excess slip removal compared to the traditional slip casting because whatever amount of slip poured into the mould will come out as the final product. Thus, mass production is guaranteed.

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References

- [1] Zainal Zakaria, Hamdzun Haron, Characterisation of local bone ash for bone china production, *Jurnal Teknologi*, Vol 66:1(2014), p. 25-34.
- [2] Alpagut Kara, Production of bone ash for manufacture of bone china, *Industrial Ceramic*, Vol.843 (1989), p.767-770.
- [3] Ahmet Capoglu, A novel low-clay translucent whiteware based on anorthite, *European Ceramic Society*, Vol.31 (2011), p. 321-329.
- [4] Ahmet Copaglu, Elimination of discolouration in reformulated bone china bodies, *European Ceramic Society*, Vol.25 (2005), p. 3157-3164.
- [5] Alpagut Kara, Ron Stevens, Characterisation of biscuit fired bone china body microstructure Part I: XRD and SEM of crystalline phases, *European Ceramic Society*, Vol.22 (2002), p. 731-736.
- [6] Abdul Rahim Mahamad Sahab, Nor Hayati Saad, Farrahshaida M. Salleh, Azlin Hamidi, Doll Said Ngah, Teng Wang Dung, Integrated process of ceramic slip using rotating technique, *Trans Tech Publications*, Switzerland (2016), Vol. 1133, p. 161-165.
- [7] Nor Hayati Saad, Abdul Rahim M. Sahab, M. Moza M. Mohtar, Farrahshaida M. Salleh, Izdihar Tharazi, Juri Saedon, Formation of

- hollow shape porcelain by using a new integrated slip rotary moulding technique, *Jurnal Teknologi*, 76:6 (2015), p. 49–53.
- [8] Abdul Rahim Mahamad Sahab, Nor Hayati Saad, Azlin Hamidi, Farrahshaida M. Salleh, Doll Said Ngah, Teng Wang Dung, High solid content slip formulation and polymer based dispersant analysis for hollow porcelain product fabrication using rotary moulding, *ICENS* (2015).
- [9] I. A.H. Al-Dawery, J.G.P. Binner, G. Tari, P. R. Jackson, W. R. Murphy, M. Kearns, Rotary moulding of ceramic hollow wares, *European Ceramic Society*, Vol.29 (2009), p. 887-891.
- [10] Ruphal Mehta, Hollow ceramic components produced by rotary moulding cut costs, *Material World Magazine*, Jan 2007.
- [11] R. J. Crawford, *Rotational Moulding of Plastics*, Wiley, 1992
- [12] Alpagut Kara, Ron Stevens, Characterisation of biscuit fired bone china body microstructure Part II: Transmission electron microscopy (TEM) of glassy matrix, *European Ceramic Society*, Vol.22 (2002), p. 737-743.
- [13] Phillippe Boch, Jean-Claude Niepce, *Ceramic Materials: Processes, Properties and Applications*, iSTE, 2001.
- [14] Yuji Hotta, Microstructural changes in sintered Al₂O₃ acid treatment of compacts produced by slip casting in gypsum molds, *Ceramic Internationals*, Vol.28 (2002), p. 593-599.
- [15] Takumi Banno, Yuji Hotta, Saburo Sano, Akhiro Tsuzuki, Kiichi Oda, Cake growth control in slip casting, *European Ceramic Society*, Vol.21 (2001), p. 879-882.
- [16] Data Sheet: Sodium Silicate Liquid Grade: 58BE (filter grade), *Concord Chemicals Corporation SDN. BHD.*
- [17] Shih-Jung Liu, Kwang-Hwa Fu, Effect of enhancing fins on the heating/cooling efficiency of rotational molding and the molded product qualities, *Polymer Testing*, Vol.27 (2008), p.209-220.
- [18] Binner, J.G.P., Mc Dermott, A.M., Yin, Y., Sambrook, R.M, and Vaidhyanathan, B. 2006. *In Situ Coagulation Moulding: A New Route for High Quality Net-shape Ceramics*. *Ceramic International*. 32: 29-35.
- [19] Yin Zhang, Deshuang Kong and Xia Feng, Fabrication and properties of porous β -tricalcium phosphate ceramics prepared using double slip-casting method using slips with different viscosities, 38 (2012), p.2991-2996.
- [20] Y. Zhang, Y. Yokogawa and T. Kameyama, Influence of powder particle size of slurries on mechanical properties of porous hydroxyapatite ceramics, *Key Eng. Mater.* 284-286 (2005), p.365- 368
- [21] Athena Tsetsekou, Christos Agrafiotis, Aggelos Miliadis, Optimization of the rheological properties of alumina slurries for ceramic processing applications Part 1: Slip-casting, *European Ceramic Society*, Vol 21 (2001), p. 363-373.
- [22] Giuliano Tari, Jose. F. Ferreira, Influence of solid loading on drying-

- shrinkage behaviour of slip cast bodies, European Ceramic Society, Vol.18 (1998), p. 487-493.
- [23] Soumen Maity and B.K Sarkar, Phase analysis and role of microstructure in the development of high strength porcelains, 24 (1998), 259-264.
- [24] Soumen Maity and B.K Sarkar, Development of high-strength whiteware bodies, 16 (1996) p.1083-1088.
- [25] Kaili Lin, Jiang Chang, Jianxi Lu, Wei Wu and Yi Zeng, Properties of β -Ca₃(PO₄)₂ bioceramics prepared using nano-size powders, 33 (2007), p.979-985.
- [26] R. Emadi, F. Tavangarian, S.I.R. Esfahani, A. Sheikhhosseini and M. Kharaziha, Nanostructured forsterite coating strengthens porous hydroxyapatite for bone tissue engineering, J. Am. Ceram. Soc. 93 (9) (2010), p.2679-2683.
- [27] K.C.B. Yeong, J. Wang and S.C. Ng. Fabricating densified hydroxyapatite ceramics from a precipitated precursor, Mater. Lett. 38 (3) (1999), p.208-213.
- [28] Hamimah Abd Rahman and Dzul Hafez Yacob, Effects of double sintering on the properties of porous ceramic.
- [29] N.J. Shaw, Densification and corsening during solid state sintering of ceramics: a review of models III. Corsening. Powder Metall. Int. 21 (5), 1989, p. 25-29.
- [30] E.A Vasilyeva, L.V Morozova, A.E Lapshin and V.G Kanakov, Ceramic materials with controlled porosity, Journal of Materials Ohysic Machanical, Vol 5 (2002), p.43-48.
- [31] L.J Fibson and M.F Ashby, Cellular solids structure and properties, Pergamon Press (1998).
- [32] S. Dhara, M. Pradhan, D. Ghosh and P. Bhargava, Nature Inspired Processing Routes for Ceramic Foams, Journal of American Ceramic Society, Vol.86 (2003), p.1645-1657.