

This article was downloaded by: [Siauliu University Library]

On: 17 February 2013, At: 06:51

Publisher: Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954

Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Advanced Composite Materials

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/tacm20>

Fabrication of Lightweight Aggregates from FRP Waste

Soo-Man Sim^a & Ki-Soo Kim^b

^a School of Materials Science and Engineering, Hongik University, Jochiwon, Chungnam 330-701, Korea

^b School of Materials Science and Engineering, Hongik University, Jochiwon, Chungnam 330-701, Korea

Version of record first published: 02 Apr 2012.

To cite this article: Soo-Man Sim & Ki-Soo Kim (2010): Fabrication of Lightweight Aggregates from FRP Waste, *Advanced Composite Materials*, 19:3, 289-297

To link to this article: <http://dx.doi.org/10.1163/092430410X504134>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.tandfonline.com/page/terms-and-conditions>

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae, and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand, or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Fabrication of Lightweight Aggregates from FRP Waste

Soo-Man Sim* and Ki-Soo Kim

School of Materials Science and Engineering, Hongik University, Jochiwon,
Chungnam 330-701, Korea

Received 7 October 2009; accepted 30 November 2009

Abstract

Lightweight aggregates were prepared from ground FRP waste, which had been decomposed in N_2 gas or air, mixed with clay as a binder and $CaCO_3$ as a foaming agent, and fired at temperatures between $800^\circ C$ and $950^\circ C$ in air. The fabrication of the lightweight aggregates was found to depend on the amount of char, $CaCO_3$, and the firing temperature. At all temperatures, the samples prepared from the FRP waste that had been decomposed in N_2 gas did not yield pore structure required for lightweight aggregates due to a relatively large amount of char. However, the sample containing a small amount of char prepared from the FRP waste that had been decomposed in air showed well-developed closed pores at $900^\circ C$ and $950^\circ C$. The addition of 5 wt% $CaCO_3$ resulted in the most uniform pore structure. As the amount of $CaCO_3$ or the firing temperature was increased, the pore size and its distribution became larger and broadened.

© Koninklijke Brill NV, Leiden, 2010

Keywords

FRP waste, lightweight aggregate, foaming agent, closed pore, firing

1. Introduction

Fiber reinforced plastics (FRPs) have been widely used in various industries such as construction, automobiles, aircraft and fishing boats because of their low weight, high strength, durability and corrosion resistance. The disposal of FRP waste, however, becomes a serious problem as the number of FRP products is increasing each year. FRP, manufactured by laminating polyester resin with glass fiber and fillers, is known as one of the most difficult materials to be recycled [1].

A number of recycling methods have been proposed and developed for FRP. There are three types of recycling: a mechanical process that produces ground FRP by reducing the size of the scrap; a thermal process that employs pyrolysis for material and energy recovery; and a chemical process that utilizes decomposition to recover monomers and fibers [1–6]. Recently, recycling of FRP waste by a cement

* To whom correspondence should be addressed. E-mail: smsim@hongik.ac.kr

Edited by KSCM

kiln has been recognized as a suitable process that supplies energy and raw materials for making cement [7]. At present the most common disposal methods are incineration and landfills because the cost of disposal of waste is relatively low [1, 8].

Incinerated or thermally decomposed products of FRP waste contain mostly glass fibers, which are coated with char [1, 9]. For the glass fibers to be recycled as reinforcement, the char must be cleaned. If the fibers are to be used in low grade insulation, the char does not need to be removed. As an alternative, the incinerated product can be used in fabrication of lightweight aggregate. The glass fiber can serve as a material for solid aggregates, and char may provide the high porosity necessary for low weight [10]. The aggregates must contain closed pores to obtain impermeability. The closed pores are formed within the solid under conditions that the glass phase is fluid enough to allow expansion of gas bubbles, but sufficiently viscous to retain the bubbles [11–13]. If char can decompose and evolve gases during a firing process, the closed pores may form in the aggregates. Otherwise, fabrication of such aggregates requires additives for control of glass viscosity and gas evolution.

In this study, lightweight aggregates were prepared using decomposed product of the scrap of glass fiber containing FRP waste. The effects of residual char and CaCO_3 , a foaming agent, on fabrication of light weight aggregates were investigated.

2. Experimental Procedure

Scraps of FRP waste taken from a disused fishing boat were thermally decomposed in a rotary furnace and used as raw material for lightweight aggregates. The FRP scraps were fed continuously into the rotary furnace at 500°C under flowing N_2 gas or air. The decomposed product containing glass fibers and char was ball-milled into powder form for one hour using a ZrO_2 ball. The powder was mixed with 2 wt% of clay as a binder to form aggregates and 2, 5 and 10 wt% of CaCO_3 (ACS grade, Aldrich Chemical Co.) as a foaming agent.

Spherical shaped samples of about 10 mm diameter were prepared using a pan-type pelletizer and were dried at 80°C for 12 h. The samples were fired in a tube furnace which had been preheated to between 800°C and 950°C in air. The samples were placed in a Pt crucible, inserted slowly into the center of the tube within five minutes, and held for 10 min.

The char content in the FRP decomposed in the rotary furnace was determined with TG/DTA (TG-DTA 2000, MAC Science) at a heating rate of $5^\circ\text{C}/\text{minute}$ in air up to 800°C . The decomposition of the char and CaCO_3 during firing was investigated by weight changes of the sample before and after firing. Apparent densities of the fired samples were measured with a He gas pycnometer (AccuPyc 1330, Micromeritics). Pore structure of the fired samples was observed with SEM (JSM-5410, JEOL). The strength of the sample was evaluated by a maximum compressive load at the point of fracture using a universal testing machine at a crosshead speed

of 2 mm/min. Average diameters of the test samples containing 2, 5 and 10 wt% CaCO_3 were 10, 11.6 and 17.1 mm, respectively.

3. Results and Discussion

Figure 1(a) is an SEM photograph of the FRP waste that was decomposed in N_2 gas, showing that char remains between glass fibers and adheres to the surface of glass fiber. The glass fibers in size of tens of μm , which were present as bundles, were unable to form aggregates. Milling of the FRP revealed that both char and glass fibers were easily ground into tens of μm and a few hundreds of μm in sizes, respectively, as shown in Fig. 1(b).

TG/DTA curves of the FRP waste decomposed in N_2 gas are presented in Fig. 2(a), showing a strong exothermic peak at 450.6°C due to the combustion of the char in air. The char content was found to be 12.1 wt%, as indicated by the weight loss. However, the decomposition in air significantly reduced the char content by 1.4 wt%. The small weight loss in Fig. 2(b) illustrates that a small amount of the char remained in the previous decomposition process.

The weight losses of the lightweight aggregates fired at various temperatures are shown in Fig. 3. Considering the weight losses of the decomposed FRP in Fig. 2 and the theoretical weight loss of 44% owing to the decomposition of CaCO_3 , the weight loss of the aggregate sample must increase in proportion to CaCO_3 content. All samples exhibited such increases in weight loss except those prepared from the FRP decomposed in N_2 and fired at 800°C .

The weight losses of the samples prepared from the FRP decomposed in air were found to be close to the expected value. For instance, the sample containing 10 wt% CaCO_3 showed the weight loss of 5.1% at 950°C , which is close to the expected weight loss of 5.6%. Since the FRP waste decomposed in air contained much less char than that decomposed in N_2 gas, the weight loss is mostly due to the decomposition of CaCO_3 . However, the weight losses of the samples prepared from

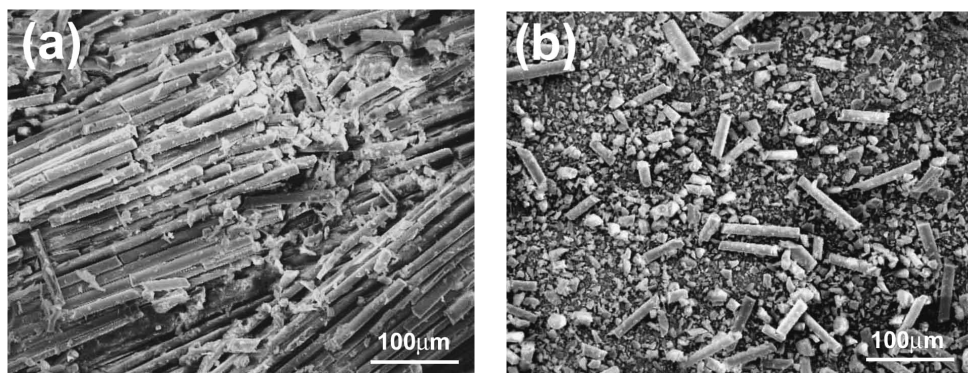


Figure 1. SEM Photographs of the FRP waste: (a) thermally decomposed at 500°C in flowing N_2 gas and (b) milled for one hour.

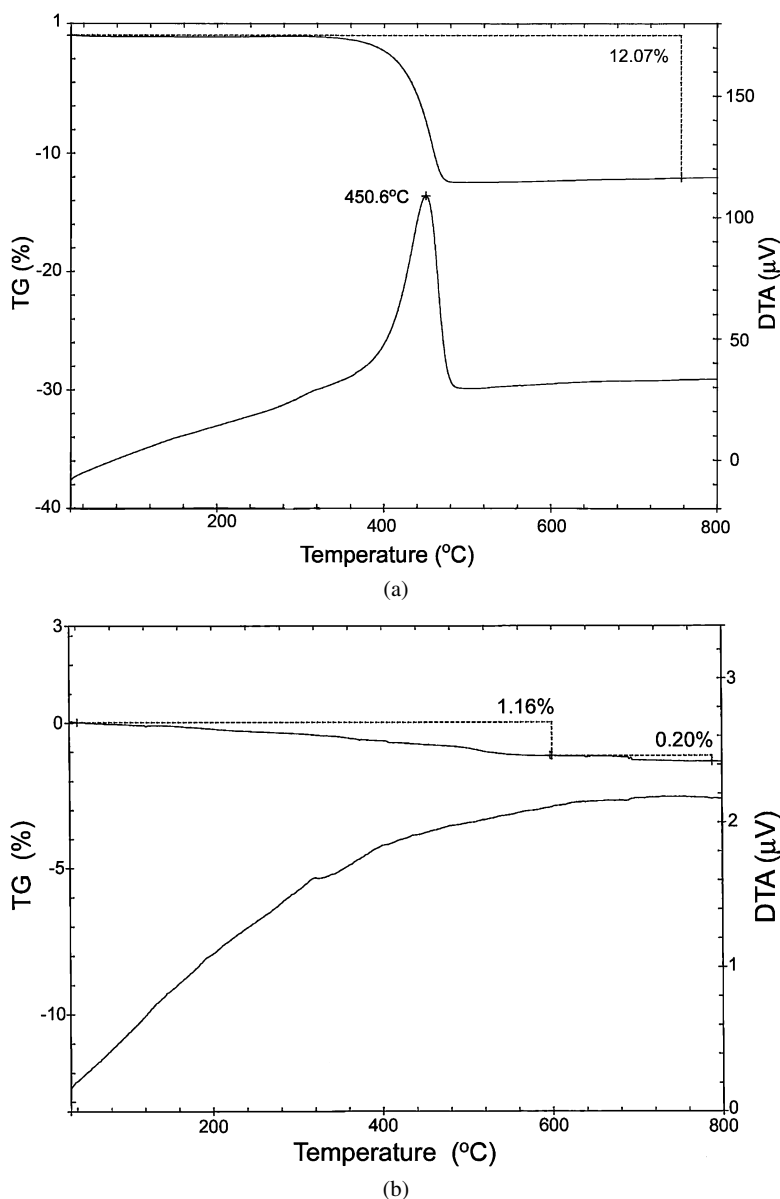


Figure 2. TG/DTA curves of the FRP scrap decomposed in flowing: (a) N_2 gas and (b) air.

the FRP decomposed in N_2 were much less than expected. The sample containing 10 wt% $CaCO_3$ yielded weight loss of 9.1% at $950^\circ C$ in contrast to the expected weight loss of 15.3%. This indicates that the combustion of the char is retarded because air flow for the combustion of the char is more difficult in aggregate form in a short firing period than in powder form. The glass particles, moreover, can shield the char against combustion when they become fluid and coalesce during

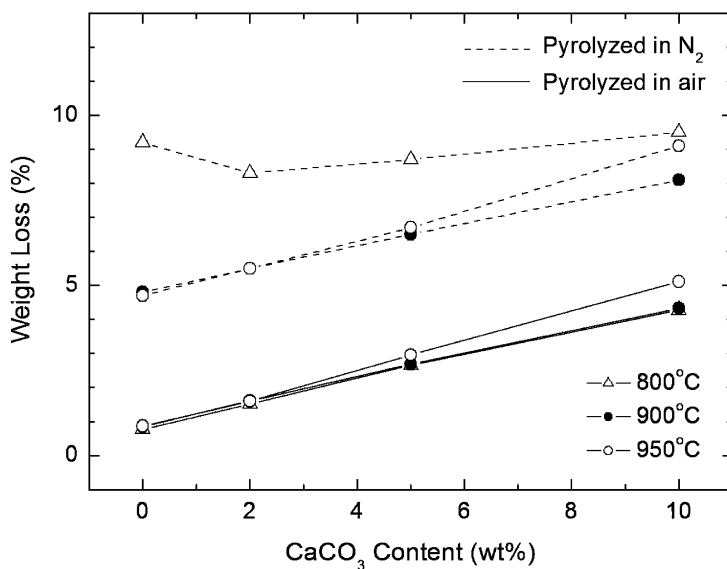


Figure 3. Weight losses of the lightweight aggregates as a function of CaCO₃ content.

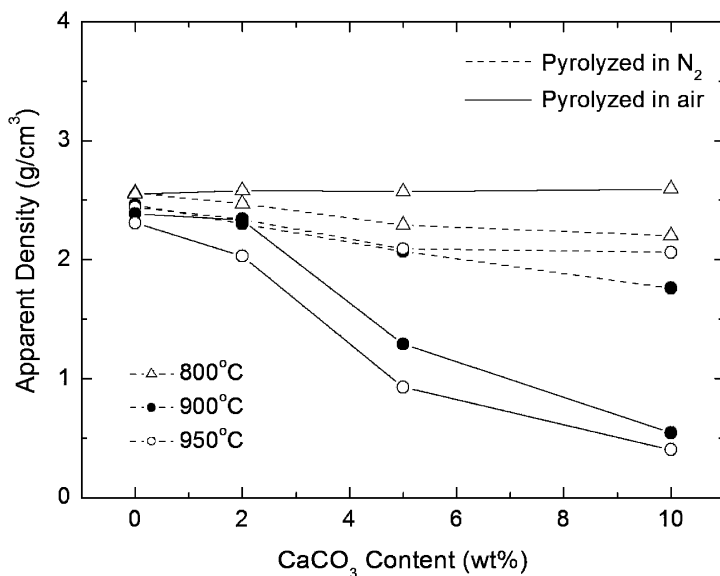


Figure 4. Apparent densities of the lightweight aggregates as a function of CaCO₃ content.

firing. If the firing temperature is too low to make the glass phase fluid, escape of gases evolved from decomposing the char will not be inhibited. Furthermore, it would result in a large weight loss as mentioned for the samples fired at 800°C.

Figure 4 represents apparent density changes in the lightweight aggregate samples fired at various temperatures. A decrease in the apparent density with the

addition of CaCO_3 is associated with an increase in closed pores inside the sample. Apparent density must decrease in indirect proportion to the volume of closed pores because the density is based on the volume including the closed pores. The samples prepared from the FRP waste decomposed in air, which contained small amounts of the char, resulted in large changes in the density, except for those fired at 800°C . Such change can be explained by the fact that a larger amount of glass phase compared to the char content effectively entraps the gas bubbles during the firing process. The addition of 10 wt% CaCO_3 exhibited the lowest density of 0.40 g/cm^3 at 950°C .

The formation of closed pores was found to depend on the firing temperature as well as the amounts of the char and CaCO_3 . The closed pores in the samples, prepared from the FRP waste decomposed in air, can be formed as long as the viscosity of the glass prevents the escape of rapidly evolving CO_2 gas during firing [11, 12]. The viscosity for the formation of the closed pores is correlated with a softening point of the glass. The glass fiber is known to have a similar composition to E-glass, which has a softening point of 840°C [14]. At 800°C , below the softening point, the glass phase is still too viscous to form gas bubbles inside the sample, which results in little changes in the apparent density. At temperatures higher than 900°C , the glass phase becomes fluid to allow expansion of gases but still viscous to retain the gas bubbles, resulting in well-developed closed pores.

For the samples prepared from the FRP waste decomposed in N_2 gas, the large amounts of the char hindered the formation of closed pores, resulting in small changes in the density with an increase in CaCO_3 content. This arises from the fact that the glass phase dispersed in the char particles could not totally prevent the escape of the gases while producing open pores instead.

The fired samples prepared from the FRP waste decomposed in N_2 gas showed no significant changes in size regardless of the amount of CaCO_3 . However, the fired samples prepared from the FRP waste decomposed in air exhibited different sizes with respect to CaCO_3 content and the firing temperature. The samples fired at 900°C and 950°C showed smooth and glassy surfaces of which sizes increased in proportion to CaCO_3 . On the other hand, the samples fired at 800°C yielded a rough surface and no changes in size, indicating that no glass phase was formed.

The effects of the addition of CaCO_3 and the firing temperature on pore structure of the lightweight aggregates can be seen in SEM photographs of Figs 5 and 6. All samples were prepared from the FRP waste decomposed in air. The samples in Figs 5 and 6 were fired at 900°C and 950°C , respectively. The addition of CaCO_3 up to 10 wt% resulted in well-developed closed pores, as discussed in Fig. 4.

The sample containing 5 wt% CaCO_3 , which was fired at 900°C , shows a relatively uniform pore size ranging from 5 to $10\text{ }\mu\text{m}$ with a few large pores as shown in Fig. 5(a). When CaCO_3 content was increased, the pore size and its distribution became larger and broadened. In Fig. 5(b), the addition of 10 wt% CaCO_3 resulted in pores in size of about $50\text{ }\mu\text{m}$ along with some small pores, as seen in the sample containing 5 wt% CaCO_3 . As the firing temperature was increased to 950°C , the

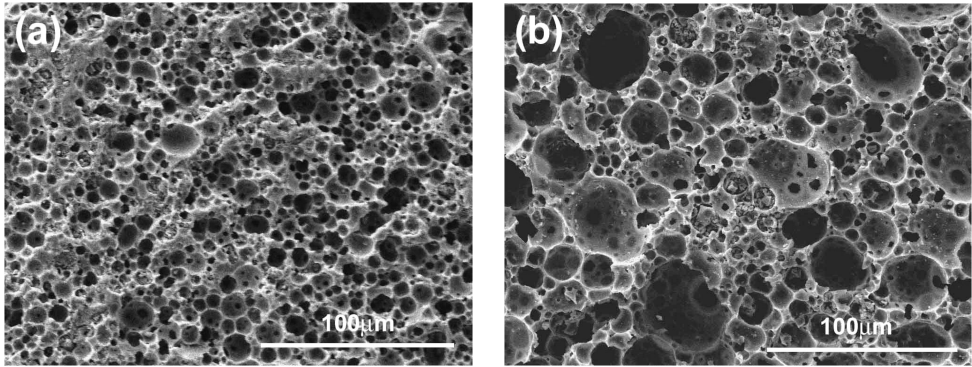


Figure 5. SEM photographs of the lightweight aggregate samples fired at 900°C: (a) 5 wt% CaCO_3 and (b) 10 wt% CaCO_3 .

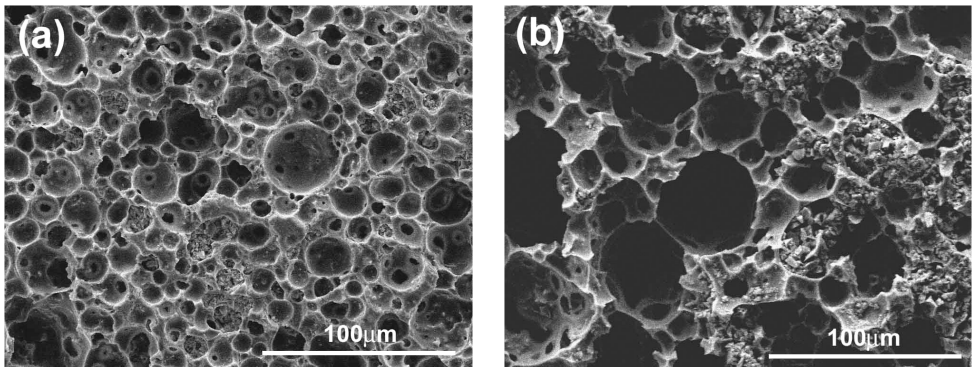


Figure 6. SEM photographs of the lightweight aggregate samples fired at 950°C: (a) 5 wt% CaCO_3 and (b) 10 wt% CaCO_3 .

glass phase became more fluid. As a result, large pores were observed as shown in Fig. 6(a). The pore sizes in the sample containing 5 wt% CaCO_3 were similar to the sample containing 10 wt% CaCO_3 at 900°C, but the sizes in the former were more uniform than those in the latter. However, the addition of 10 wt% CaCO_3 exhibited larger pores in size ranging from 50 μm to 80 μm, as shown in Fig. 6(b). Small particles filling some pores were the fragments of the sample which had been produced in the cutting process.

The strength of the aggregate sample, prepared from the FRP waste decomposed in air and fired at 900°C, was evaluated by a maximum compressive load at the point of fracture. Figure 7 shows that the maximum load was rapidly decreased from 2922 N for the sample containing 2 wt% CaCO_3 to 83 N for the samples containing 10 wt% CaCO_3 . Experimentally, it is known that the strength of porous ceramics is decreased exponentially with porosity [15]. A decrease in the strength of the samples with the addition of CaCO_3 coincides with changes in the apparent density, which are related to the volume of the close pores as shown in Fig. 4.

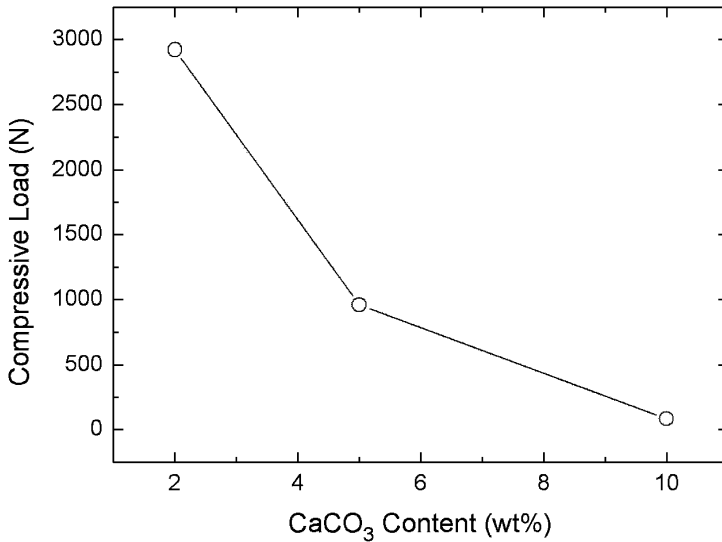


Figure 7. Maximum compressive load for fracture of the lightweight aggregates fired at 900°C as a function of CaCO₃ content.

4. Conclusion

Lightweight aggregates were prepared from ground FRP waste, which had been decomposed in N₂ gas or air and fired at temperatures between 800°C and 950°C in air. The fabrication of the lightweight aggregates was found to depend on the amount of char, CaCO₃, a foaming agent and the firing temperature. Formation of closed pores in the aggregate samples could be confirmed by the apparent density indirectly proportional to the amount of CaCO₃.

The samples prepared from the FRP waste decomposed in N₂ gas did not yield pore structure required for lightweight aggregates due to a relatively large amount of char. However, the sample containing a small amount of char, prepared from the FRP waste decomposed in air, showed well-developed closed pores at 900°C and 950°C. The samples containing 5 wt% CaCO₃, fired at these temperatures, yielded the most uniform pore structure. As the amount of CaCO₃ or the firing temperature was increased, the pore size and its distribution became larger and broadened.

Acknowledgement

This work was supported by 2008 Hongik University Research Fund.

References

1. A. Conroy, S. Halliwell and T. Reynolds, Composite recycling in the construction industry, *Composites: Part A* **37**, 1216–1222 (2006).
2. S. J. Pickering, Recycling technologies for thermoset composite materials — current status, *Composites: Part A* **37**, 1206–1215 (2006).

3. W. Dang, M. Kubouchi, H. Sembokuya and K. Tsuda, Chemical recycling of glass fiber reinforced epoxy resin cured with amine using nitric acid, *Polymer* **46**, 1905–1912 (2005).
4. A. Kamimura, K. Yamada, T. Kuratani, Y. Taguchi and F. Tomonaga, Effective depolymerization waste FRPs by treatment with DMAP and supercritical alcohol, *Chem. Lett.* **35**, 586–587 (2006).
5. T. Iwaya, S. Tokuno, M. Sasaki, M. Goto and K. Shibata, Recycling of fiber reinforced plastics using depolymerization by solvothermal reaction with catalyst, *J. Mater. Sci.* **43**, 2452–2456 (2008).
6. M. Goto, Chemical recycling of plastics using sub- and supercritical fluids, *J. Supercrit. Fluids* **47**, 500–507 (2009).
7. Recycling Threat to Europe's Composites Industry, *Reinforced Plastics*, Comment, September (2006).
8. Facing Up to the Recycling Challenge, *Reinforced Plastics*, June 22–26 (2001).
9. A. M. Cunliffe and P. T. Williams, Characterization of products from the recycling of glass fibre reinforced waste by pyrolysis, *Fuel* **82**, 2223–2230 (2003).
10. A. Mueller, S. N. Sokolova and V. I. Vereshagin, Characteristics of lightweight aggregates from primary and recycled raw materials, *Constr. Build. Mater.* **22**, 703–712 (2008).
11. J. E. Conley, Production of lightweight concrete aggregates from clays, shales, slates and other materials, *US Bur. Mines Reports* **4401**, 121–128 (1948).
12. L. J. Gibson and M. F. Ashby, *Cellular Solids: Structure and Properties*. Pergamon, Oxford, UK (1998).
13. E. C. D. Lima, P. P. C. Sartoratto, A. M. Ayres and S. B. Oliveira, Aluminum/sodium phosphate-based closed cell ceramic foams, *J. Non-Cryst. Solids* **279**, 60–71 (2001).
14. Technical Data, LCS Laboratory Consulting Service, GmbH, <<http://www.labconservice.de>>, cited 20 Aug. (2009).
15. W. D. Kingrey, H. K. Bowen and D. R. Uhlmann, *Introduction to Ceramics*, 2nd edn. Wiley, New York, USA (1976).