Processing of Composite Material Using Industrial Waste Aluminum Sludge

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SUMMARY: The application of BMC composite material using aluminum sludge from the point of identifying more effective uses was attempted. BMC is composed of glass fiber, unsaturated polyester and filler. Aluminum sludge was applied as the BMC filler of the composite material used for the injection molding and the injection compression molding. With a sludge content of 40% and the glass fiber content maintained at a constant 10%, the tensile strength of the product showed the highest value. From the comparison of sludges at three separate levels of heat treatment, the sludge structure fired at 773K crystallized into $\gamma$ alumina and the product containing $\gamma$ alumina showed the highest mechanical strength.


1. INTRODUCTION

Aluminum sludge is a precipitation yielded from the acid/alkali neutralizing process of waste fluid which is derived from the surface treatment of aluminum sash. Aluminum sludge is designated as industrial waste which should be disposed of at areas specified by public institutions. We have investigated the possibility of recycling the sludge into a useful resource from the standpoint of ensuring the safety and protection of the environment. Therefore, the sludge underwent heat treatment and was applied as a ceramics material for injection molding as its most valuable use. As a result, the sludge was transferred into $\alpha$ alumina structure, and it was found that ceramics injection molding products have sufficient strength(1). However, a big problem of sulfurous acid gas being generated occurred between temperatures of 873 and 1073K during the process of firing the sludge. Because the sulfurous acid gas is harmful, proper equipment is required in order to render it harmless.

In this research, we attempted the application of BMC (Bulk Molding Compound) composite material using aluminum sludge for the effective use below the temperatures of which the sulfurous acid gas was generated. BMC is a common material used in the manufacturing of FRP (Fiber Reinforced Plastics). BMC offers the designer a very useful combination of mechanical, electrical, and thermal properties (2). The sludge performed the role of the BMC filler. BMC composite material is composed of glass fiber which plays the role of enforcement, unsaturated polyester which forms the molding, and filler. The purpose of the filler is to save resin and add product characteristics. First, as a step of our experimental method, a molding machine was designed on an experimental basis. Next, while varying the content of aluminum sludge between the ranges of 10% and 40%, the products were investigated in terms of mechanical strength and quality. In order to determine the optimum heat condition, the sludge was treated using three types of heat treatment. In the first case, the water included with the sludge was evaporated at temperatures of 373K and above. In the second case, the $\gamma$ alumina sludge was treated below 873K which had no occurrence of sulfurous acid gas. In the third case, the sludge was fired at 1573K and transferred into $\alpha$ alumina structure which is a common commercial material.
2. EXPERIMENTAL MATERIAL AND METHOD

2.1 Aluminum sludge

First, the aluminum sludge discharged from the factory was sufficiently dried by sun light and then three types of heat treatment were applied at the temperatures 423, 773, and 1573K. The temperature 423K was set up merely for the complete evaporation the water included with the sludge. 773K was prepared for the sludge treated with $\gamma$ alumina to avoid the occurrence of sulfurous acid gas. And 1573K was set up for the sludge treated with the crystal structure of common ceramics material $\alpha$ alumina. These sludges were fired at each temperature for two hours and then cooled gradually in the furnace. After the heat treatment, the sludges were milled for eight hours and then used for experimentation.

2.2 Experimental method

Prior to being formed by injection molding, the BMC composite material of chopped strand type glass fiber, unsaturated polyester and sludge filler was premixed in a two-screw type kneading machine. Table 1 shows the ingredient ratio of BMC. Because the BMC premixture has a high viscosity, the injection instrument was specially designed with the purpose of avoiding stoppage in the barrel, sprue, runner and cavity of mold. Particularly, the plunger type injection instrument was adopted in order to prevent the breakage of glass fiber in the barrel. Fig.1 shows the instrument for injection molding and injection compression molding. The BMC loaded into the hopper was pushed by a plunger to the nozzle, sprue and mold. As unsaturated polyester is a thermosetting resin, two heaters were set up behind the mold separately. Table 2 shows the injection molding condition. The estimation of the molding product was carried out by tensile test, bending test, hardness test and comparison of shrinkage ratio.

<table>
<thead>
<tr>
<th>Table 1 Ingredient ratio.</th>
<th>Table 2 Condition of injection molding.</th>
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<tbody>
<tr>
<td>Material</td>
<td>$I$</td>
</tr>
<tr>
<td>Aluminum sludge</td>
<td>10</td>
</tr>
<tr>
<td>Unsaturated polyester</td>
<td>79</td>
</tr>
<tr>
<td>Glass fiber</td>
<td>10</td>
</tr>
<tr>
<td>Zinc strand</td>
<td>0.7</td>
</tr>
<tr>
<td>Perbutyl Z</td>
<td>0.3</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
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Fig.1 Plunger type injection-molding instrument.

3. EXPERIMENTAL RESULT AND DISCUSSION

3.1 Effect of sludge content on the mechanical property of injection product

Because the sulfurous acid gas from the sludge was generated between temperatures of 873K and 1073K, the sludge underwent heat treatment at 773K which had no occurrence of
sulfurous acid gas. While varying the content of sludge in the BMC, the products were investigated in terms of mechanical strength and quality. As the BMC is composed of unsaturated polyester, glass fiber as an enforcement material, and filler, the sludge was used as a filler. However, when the sludge content reached 43% in the BMC with a constant glass fiber content maintained at 10%, stoppage of the BMC occurred at the nozzle and the injection molding could not be performed. Therefore, the sludge content was varied between the range of 10 and 40%, and the effect of the sludge content on the mechanical strength of product was investigated.

Fig.2 shows the relation between the sludge content and tensile strength of the product. Within the range of 10 and 30%, the tensile strength remained the constant, however, the tensile strength at the sludge content of 40% showed the highest strength. Fig.3 shows the microphotographs of the fracture surface of the tensile test through the scanning electron microscope (SEM). It was observed that within the sludge content range of 10 and 30%, the glass fibers were scattered uniformly. As the chopped strand glass fibers at the sludge content 40% were loosened uniformly, it could be considered that the tensile strength at the sludge content of 40% showed the highest value. Next, the relation between the sludge content and the bending strength of the product is shown in Fig.4. The bending strength shows a constant tendency regardless of the variation of sludge content between 10 and 40%. Fig.5 shows the photomicrographs of the initial rising fracture area in the bending test. Each fracture
morphology appears to be similar at the edge point, therefore, there is no clear difference among the sludge contents of 10 to 40%. Fig.6 shows the relation between the sludge content and Rockwell hardness of product. The product hardness shows a constant tendency regardless of the sludge content. Fig.7 shows the relation between the sludge content and the product density. As the sludge content increases from 10 to 40%, the density increases gradually and finally reaches 1.50Mg/m$^3$ at the sludge content of 40%. Fig.8 shows the relation between the sludge content and shrinkage ratio of the product. As the sludge content increases, there is a tendency that the shrinkage ratio of the product decreases gradually. This phenomenon is attributed to the effect of the sludge powder particles curbing the shrinkage of unsaturated polyester due to chemical reaction. Therefore, high sludge content in the BMC contributes to high accuracy of the dimensions of the mold.

### 3.2 Effect of heat treatment to sludge on mechanical property of product

X-ray analysis of sludge was carried out in order to investigate the crystal structure by changing the firing temperature. It was obvious from the diffraction patterns shown in Fig.9 that the structures of sludge at each firing temperature are baimaite at 423K, $\gamma$ alumina at 773K and $\alpha$ alumina at 1573K. The grain size distribution measured against each sludge is shown in Fig.10. The grain size diameter of the $\gamma$ alumina fired at 773K was 6.58 $\mu$m which was the smallest. Referring to the previous experimental results, the high strength BMC was mixed with the ingredient ratio of a 40% sludge content and a maintained glass fiber content of 10%, and the effect of firing temperature to the sludge on mechanical strength was investigated. Fig.11 shows the relation between bending and tensile strength of the product and the firing temperature of the heat treatment. The bending strength and tensile strength shows the same tendency with both strengths of the product fired at 773K showing the highest value. Fig.12 shows the microphotograph of the fracture surface in a tensile test. In the case of sludge fired at 773K, the chopped strand glass fibers in the product were loosened and scattered uniformly due to the finest particle size. Fig.13 shows the microphotograph of the fracture surface in a
bending test. Although there is no difference in the state of glass fiber at the edge area which the initial crack occurs, the fracture surface at 773K was observed as having a large amount of flat surface area due to strong adhesion between the sludge powder particles and unsaturated polyester. Because the total surface area increase with the number of sludge particles due to

![Fig.9 X-ray diffraction patterns of aluminum sludge fired at 523K, 773K, 1573K, respectively.](image)

![Fig.10 Grain size distribution of aluminum sludge fired at 523K, 773K, 1573K, respectively.](image)

![Fig.11 Comparison of tensile bending of strength of product.](image)
their small size, the sludge particles were fixed firmly to the unsaturated polyester. Therefore, the strength of the product fired at 773K was improved drastically. Fig.14 shows the relation between the firing temperature and the Rockwell hardness. The Rockwell hardness of the product fired at 773K shows the highest value which is attributed to the dense filling which shortens the distance between grain particles. Fig.15 shows the relation between the shrinkage of product and firing temperature. The shrinkage of product fired at 773K shows the lowest value due to the small size of sludge scattered uniformly which affects the prevention of the shrinkage of polyester resin by chemical reaction.

3.3 Comparison of injection molding and injection compression molding

The difference between the injection molding and injection compression molding is the method of pressing the material. In the case of the injection molding, the material is filled and pressed into the closed mold using high injection pressure. Conversely, with the injection compression molding, the mold is left slightly separated, maintaining a small clearance, and the material is filled using low injection pressure. The mold is then firmly closed using high compression. Particularly, in the case of high viscosity BMC material, high injection pressure is required for filling and pressing with injection molding. On the other hand, with the injection compression molding, only filling is required and therefore low pressure is sufficient. Accordingly, injection compression molding is considered suitable for BMC material. Both method were investigated for tensile strength and bending strength of the product. The tensile strength for injection molding was 38MPa, while the injection compression molding showed a value of 36MPa which is slightly lower. The bending strength values were 60MPa for injection molding and 72MPa for injection compression molding which is 20% higher. This improvement of bending strength on injection compression molding is dependent upon the uniform glass fiber scattering by press the mold uniformly. Conversely, with the injection molding, the reduction of bending strength is attributed to the orient of the glass fiber distribution in the BMC which is highly anisotropic due to the occurrence of high pressure from the sprue area.

From the comparison of measuring the thickness distribution (64x160x4) of the product with a thickness of 4mm for both methods, the dimensional error of thickness was plus-minus 0.125mm for the injection molding and plus-minus 0.013mm for injection compression molding. Accordingly, injection compression molding has high dimensional accuracy for transcribing the mold to the product.

4. CONCLUSIONS
From the point of a highly valuable use of aluminum sludge of industry waste, sludge was applied as a filler of BMC composite material for injection molding and injection compression molding. As a result, we were able to accomplish the following:

(1) While varying the content of aluminum sludge between the ranges of 10 and 40% and maintaining the glass fiber content at a constant 10%, the product were investigated in terms of mechanical strength of quality. The tensile strength of the product with a sludge content of 40%, showed the highest value and the shrinkage ratio of the product showed the lowest value.

(2) From the comparison of sludges at three separates levels of heat treatment (523K, 773K and 1573K), the product fired at 773K in order to crystallize it into \( \gamma \) alumina structure showed the highest mechanical strength and the lowest shrinkage. The cause is attributed to the finest particle size of \( \gamma \) alumina, which is superior for cohesion because of the increased contact surface area between \( \gamma \) alumina particles and unsaturated polyester.

(3) In the case of injection compression molding using \( \gamma \) alumina sludge fired 773K with a sludge content of 40%, the product showed higher bending strength and higher dimensional accuracy than the injection molding product. From these results, the injection compression molding is considered to be a more suitable processing for using aluminum sludge as a filler of BMC composite material.

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REFERENCES
