

EFFECT OF DIE COATED WITH HEAT INSULATION ON SURFACE ROUGHNESS IN THERMOSET COMPRESSION MOLDING

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ABSTRACT

Carbon fiber reinforced plastics (hereinafter referred to as "CFRPs") have been adopted for the outer panels of automobiles, such as hoods and roofs, for weight reduction and design. To adopt CFRPs for outer panels, they are required to have equivalent surface smoothness to metals and need surface polishing after molding. Thus, in order to obtain a surface as smooth as possible in molding and to shorten a polishing time, in this study, we examined whether surface irregularities due to shrinkage of the resin between the fibers can be reduced and, in consequence, surface smoothness can be enhanced by applying a heat -insulating coating to the surface of the die for improving surface quality in methods that impregnate a resin into fabrics, such as carbon fibers.

1 INTRODUCTION

In recent years, the application of composite materials to automobiles has been gradually increasing in response to the need for weight reduction. CFRPs can reduce weight by approximately 50% compared with steel, and glass fiber reinforced plastics (hereinafter referred to as "GFRPs") have also been adopted for outer panels because of the degree of freedom of design. CFRPs and GFRPs are used in a wide variety of parts for automobiles, including outer panel parts, such as hoods and roofs, and interior decorative parts. In addition, kenaf and other types of natural fibers are also used in some parts to achieve carbon neutrality, which is currently attracting wide attention.

Surface quality is an issue with the outer panel parts using CFRPs or other composite materials. Plain-woven and twill-woven fabrics are widely used because of their design. The resin between the fibers shrinks, resulting in micro-irregularities on the surface. To eliminate irregularities, the process of applying and polishing the resin is repeated many times. Surface irregularities are attributable to materials and molding conditions, such as the state of the fiber weaving, shrinkage of the resin, and insufficient pressure inside the die. Previous studies using thermosetting materials have reported on the optimization of molding conditions for preventing shrinkage on curing of the material with the aim of improving surface quality in resin transfer molding (hereinafter referred to as "RTM") [1] and on molding conditions and surface roughness in open molding of GFRPs [2]. In RTM and wet compression molding (hereinafter referred to as "WCM"), which have been representative methods for manufacturing automotive parts in recent years, the temperature of the die is kept higher than that of the resin before the resin is injected into the die. The viscosity of the resin injected into the die temporarily decreases, before increasing as the resin becomes gelled, and the curing reaction of the resin begins rapidly. Thus, studies were conducted to elucidate the relationship between the degree of curing and the shrinkage rate in the curing reaction, as well as between the shrinkage rate and the curing temperature [3] [4], and it was found that the material being cured shows a rapid temperature change from approximately 100°C to 150°C until it becomes cured. Moreover, a rapid temperature change also occurs during injection molding of a thermoplastic resin when the molten resin is cooled inside the die. In consequence, the resin is rapidly cooled on the surface of the die and becomes less flowable and transferrable, which adversely affects the appearance quality. Some study reports and application cases have demonstrated an improvement in surface quality in injection molding by applying a heat -insulating coating to the surface of the die to prevent weld lines [5] [6]. In this study we examined if surface roughness, which affects surface quality, can be reduced by using a die with heat -insulating coating for improving surface quality in compression molding of thermosetting materials.

2 EXPERIMENTAL METHODS

2.1 Materials for the experiment

A 3K plain-woven fabric with a density of 198 g/m2 was used for carbon fibers, and two types of epoxy resins (bisphenol A type epoxy resin and polyamine as a hardener) with different curing rates were used for resins. Resin A took approximately 200 seconds to cure at a mold temperature of 100°C. Resin B took 400 seconds to cure, or approximately twice longer than Resin A. Furthermore, the gelling time of Resin A and that of Resin B were 34 seconds and 60 seconds, respectively, at a temperature of 120°C.

2.2 Die and the molding method

The experimental mold is shown in Figure 1. Chromium plating and ceramic coating were performed on the molding face of the upper die. The thickness of the chromium plating was 2 μ m, and the thickness of the ceramic coating was 500 μ m and 700 μ m. For the ceramic coating, an acrylic resin containing ceramic particles was applied to the mold and sintered, and the surface was smoothed by polishing. In addition, temperature and pressure sensors were mounted in the central part of the molding face of the upper die to measure the temperature and pressure during molding. A 20-ton compact press was used as a pressing machine. The molding procedure started by cutting a plain-woven fabric into pieces measuring 200 x 200 mm and placing four pieces on top of one another to make the base material. The base material was placed inside the die and an epoxy resin mixed with a curing agent at 50°C was poured over it. The curing time from die clamping to the release of the base material from the die was set to two levels, 5 minutes and 10 minutes. The temperature of the die was kept at 100°C. The experimental conditions are shown in Table 1.



Figure 1: Experimental die and test pieces

Experimental conditions	Level 1	Level 2	Level 3
Material type	A (fast curing)	B (normal)	_
Material temperature ^{$\times 1$}	50°C	_	_
Coating	Not applied	Applied (500 μm)	Applied $(700 \mu m)$
Die clamping pressure	20tons	_	_
Die temperature	100°C	_	—
Die clamping rate	2mm/sec	—	—

*1: material temperature is the temperature at which an epoxy resin and curing agent are mixed and stirred.

Table 1: Experimental conditions

2.3 Analysis method

After molding the test pieces, the surface roughness of the central part of the test pieces was measured using a laser microscope. In addition, to investigate the material characteristics that affect surface irregularities, we measured the shrinkage-on-curing rates of the two resins, Resin A and Resin B.

3 EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Surface roughness result

The surface state of the test pieces observed through the laser microscope and the measured surface roughness are shown in Figure 2. It can be observed that the test pieces consist of plain-woven fibers and only the areas with resin between the fibers are concave.



Figure 2: Observation of the surface state and measurement of surface roughness

The results for surface roughness are shown in Figure 3. In a comparison between Resin A and Resin B with different curing rates, the surface roughness of Resin B, which was slow in curing, was 14 to 44 μ m, whereas that of Resin A, which cured fast, was 38 to 60 μ m, which was higher than that of Resin B. Regarding the effect of the mold coating, the surface roughness of Resin A, which cured fast, was reduced by the coating. Resin B, which was slow in curing, showed a higher value of surface roughness with the coating than without the coating



Figure 3: Surface roughness

The results for the die clamping times are shown in Figure 4. The surface roughness of Resin A, which cured fast, was slightly reduced by changing the mold clamping time from 5 minutes to 10 minutes, but the surface roughness of Resin B did now show a significant change although the mold clamping time was changed.



Figure 4: Relationship with the mold clamping time *Roughness data (waveform)

The thermal conductivity of the heat -insulating coating used in the present study was measured. It was measured by the laser flash method, in which a coating is applied to a test material made of the same metal as the mold, it is heated by irradiating with laser beams on one side, and the change in temperature on the other side is measured. The results of the measured thermal conductivity show that it was 54 W/m \cdot K without the coating but significantly reduced to 4 W/m \cdot K with a ceramic coating of 700 µm.

The effect of the coating on surface roughness, which was the objective of the present study, was confirmed with Resin A, which cured fast. This is probably because the heat transfer from the mold to the resin was moderated by the coating and the resin flowed more smoothly as the mold clamped, resulting in the reduction of the surface irregularity. In contrast, the coating was not considered to be effective for Resin B, which was slow in curing, because of its peculiar property of gradual curing. Furthermore, no significant difference was observed in this effect between the coating thicknesses of 500 μ m and 700 μ m.

3.2 Temperature and pressure during molding

We studied the temperature and pressure changes during molding. As shown in Figure 1, temperature and pressure sensors were embedded in the central part of the upper die. The waveforms of temperature and pressure for Resin A are shown in Figure 5; the graphs in the upper panel are waveforms for the clamping time of 5 minutes and the graphs in the lower panel are waveforms for the clamping time of 10 minutes. The waveforms of temperature and pressure for Resin B are shown in Figure 6, similar to Figure 5. From a comparison between the values with and without the coating marked with the red circles in Figure 5, it is clear that without the coating, the temperature inside the mold temporarily decreased by approximately 20°C because the temperature of the resin was lower than that of the mold. In contrast, with the coating, the temperature reduction was modest, and the conduction of heat from the mold was moderated by the coating; 50 seconds later, the temperature of the mold was the same both with and without the coating.

When it comes to the behavior of the pressure sensor in contact with the mold, the pressure was approximately 4 MPa without the coating, 1 MPa with a coating thickness of 500 μ m, but undetectable with a coating thickness of 700 μ m. Without the coating, the pressure sensor was in the same position as the mold face, but with the coating, the behavior of the pressure was attributable to the application of

the coating over the pressure sensor. The pressure increased just after the mold clamped, but then gradually decreased, showing that the curing and shrinkage of the resin had progressed.

In addition, in terms of the difference in the material between Resin A and Resin B, the pressure inside the mold was approximately 4 MPa with Resin A but lower than that, or approximately 3 MPa, with Resin B with a slow curing rate, probably because Resin B cured gradually.



Figure 6: Changes in temperature and pressure (Resin B)

3.3 Shrinkage-on-curing rates of resins

Considering that surface irregularities would also affect the shrinkage on curing of the resin between the fibers, we measured the shrinkage-on-curing rate of the resin alone. We used the Custron device from AcroEdge Co.,Ltd and continuously measured shrinkage by changing the thermal profile. The measuring equipment and the method are shown in Figure 7. The resin was set in 50-cc sample holders, and the shrinkage-on-curing rate of the resin was measured using a laser displacement meter while increasing the temperature. The temperature profile inside the measuring equipment was set to two levels, 80°C and 120°C.



Figure 7: Method for measuring the shrinkage on curing of the resin

The results of the shrinkage-on-curing rates for Resin A and Resin B are shown in Figure 8. As soon as the resin was set in the measuring equipment, its volumetric shrinkage rate became negative, indicating that the resin had expanded because of its curing reaction. Then, as shrinkage on curing progressed, the volumetric shrinkage rate became positive and remained constant. The volumetric shrinkage rate of Resin A, which cured fast, was approximately 4% at 80°C and approximately 1% at 120°C. In contrast, the volumetric shrinkage rate of Resin B, which was slow in curing, was approximately 6% at 80°C and approximately 2.8% at 120°C. The resin that cured fast showed a lower shrinkage-on-curing rate.



Figure 8: Comparison of the shrinkage-on-curing rate between Resin A and Resin B

Next, using Resin B, which was slow in curing, the volumetric shrinkage rate was evaluated with stepwise change in the temperature profile inside the measuring oven by increasing the temperature from 80°C to 120°C after 2 minutes and after 3 minutes. The results of the volumetric shrinkage rates are shown in Figure 9. Immediately after the temperature was increased from 80°C to 120°C, the volumetric expansion rate became negative and the resin was in the process of shrinkage on curing, implying that the reaction was activated by the temperature increase.



Figure 9: Step-wise temperature rise

When the results of the shrinkage on curing and surface roughness of the abovementioned resins were compared, the surface roughness of Resin A having a fast curing rate was higher than that of Resin B as shown in Figure 3, which was contrary to the shrinkage-on-curing rates of the resins. Irregularities of the resin between the fibers are attributable partly to the movement of the fibers by the resin during molding.

4 CONCLUSIONS

In the process of improving the outer surface quality of composite materials, we confirmed that the effect of the mold coating on surface roughness, which is an indicator of irregularities between fibers, depends on the characteristics of the materials. This was the objective of the present research. The surface roughness of the resin that cured fast could be slightly reduced by die coating, but it was not at all effective with the resin that was slow in curing. Meanwhile, in the evaluation of the material shrinkage rate, we found that the resin that cured fast showed a low shrinkage-on-curing rate and that the shrinkage-on-curing rate decreased as the curing temperature increased. Regarding the difference between the surface roughness and shrinkage on curing of the resins, the resins were cured by direct contact with the die in the surface roughness experiment, but the shrinkage on curing of the resins was evaluated in the temperature environment of the evaluation equipment and the transfer rate of the actual mold temperature to the resin may therefore be different from that seen in the evaluation. Thus, we will continue the investigation.

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