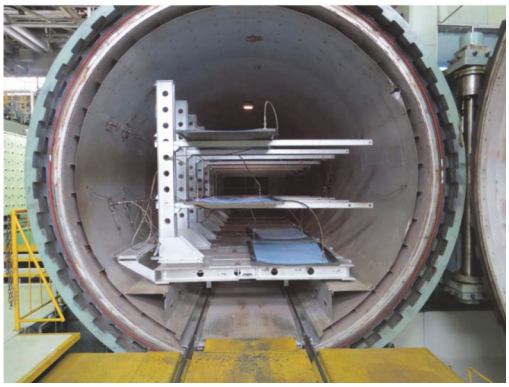
Improvement of Development Process for Thermosetting Composites Parts by Improving Accuracy of Curing Analysis



KAZUKI NOMA[*1] MASAMI KAMIYA[*1]

Light-weight and high-strength thermosetting composite materials have been increasingly applied mainly to aircraft applications. However, if the temperature conditions during the curing of the composite material are not correct, the mechanical properties can deteriorate, so the setting of the correct curing conditions for each part configuration is necessary. Generally, in the case of composite parts for aircraft, the curing conditions were set through about 5 to 10 part temperature measurement tests, and the reduction of the test cost was a challenge. Therefore, to improve the curing conditions setting process, curing analysis that can predict composite part temperature was applied and its accuracy was enhanced.

1. Introduction

Light-weight and high-strength thermosetting composite materials have been applied to a wide range of applications including aircraft. Generally, such materials are cured under high temperature and high pressure using an autoclave, but the mechanical properties change according to the temperature conditions in the curing process, so it is necessary to set the correct curing conditions for each part configuration. However, in the case of thick composite parts applied to aircraft structures, since the amount of reaction heat during the curing of the resin is large, the internal temperature increases and tends to deviate from the required value. As shown in **Figure 1**, it is necessary to set the correct curing conditions to satisfy the required part temperature, but generally in the case of composite parts for aircraft, the cost is significant because curing conditions are set through roughly 5 to 10 part temperature measurement tests.

To solve this problem, we have been promoting the reduction of the curing conditions setting cost by applying curing analysis that can predict part temperature during curing in an autoclave.[(1)] For the purpose of improving the accuracy of the curing analysis of thermosetting composite materials for aircraft, we acquired the precise thermophysical properties for analysis and verified the accuracy of the analysis. This paper reports the results thereof.

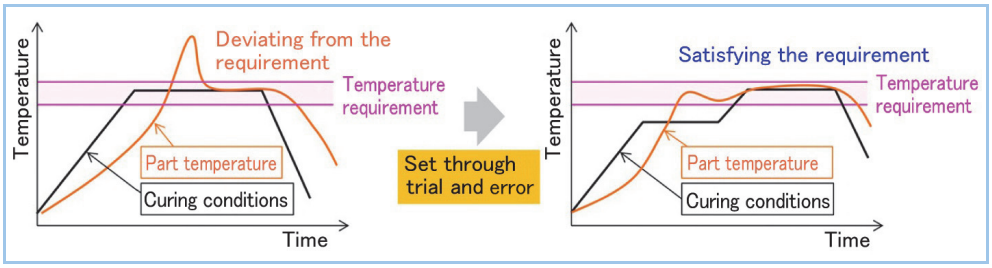


Figure 1 Curing conditions setting
An example of the curing conditions is shown. Under the curing conditions in the figure on the left, the part temperature deviates from the requirement. It is necessary to satisfy the requirement by making temperature holding steps in the course of curing as shown in the figure on the right.

*1 Manufacturing Technology Research Department, Research & Innovation Center

2. Curing analysis of thermosetting composite material

For the curing analysis, FEM analysis modeling software COMPRO-2D[2] (produced by Convergent Manufacturing Technologies) was used. Analysis can be made in consideration of the curing reaction behavior of the composite material in addition to transferring heat in the autoclave to the composite material part.

Figure 2 depicts the curing analysis workflow. After the thermophysical properties (cure kinetics, specific heat, and thermal conductivity) of the material are input, the boundary conditions (heat transfer coefficient in autoclave) are set, and the curing conditions are input. As the analysis result, the part temperatures are output, so the curing conditions satisfying the requirements can be examined, which is effective for the reduction of the test cost.

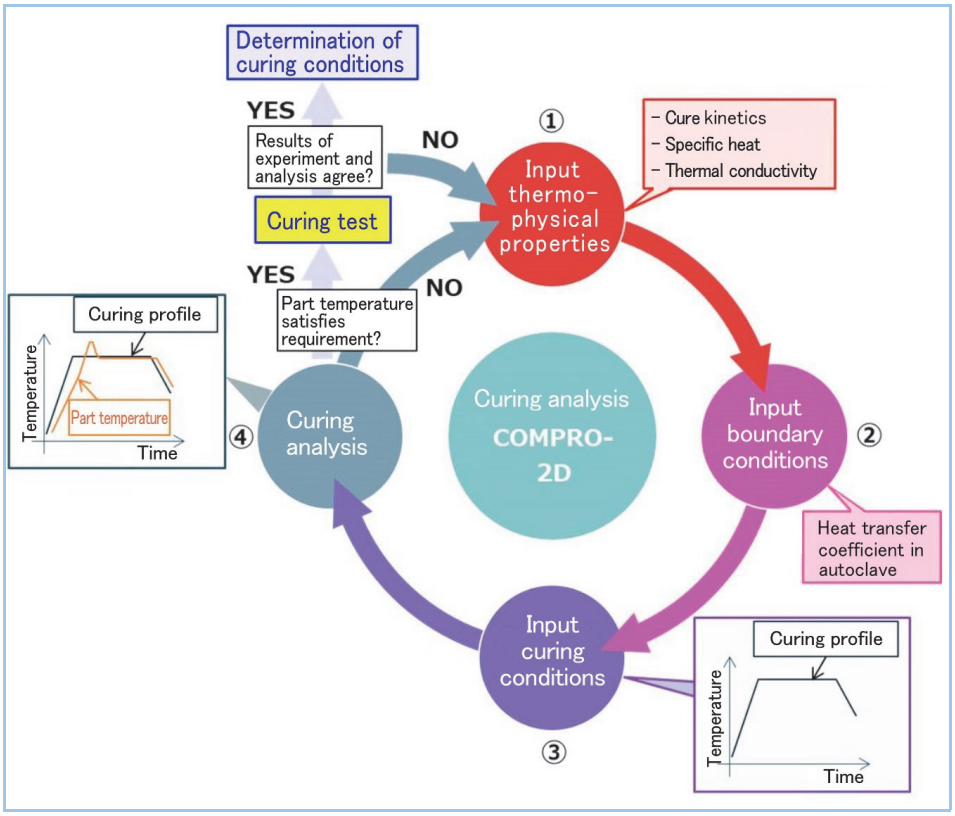


Figure 2 Curing analysis workflow
The curing analysis workflow is shown. By inputting the thermophysical properties, boundary conditions, and curing conditions, the analysis results of the part temperature is output. When the part temperature satisfies the requirement, the curing conditions are determined to be appropriate, and if not, the input conditions need to be reviewed again.

3. Accurate acquisition of thermophysical properties

For the purpose of improving the curing analysis accuracy of thermosetting composite materials, the thermophysical properties of the composite material were acquired with high precision. The cure kinetics and specific heat were derived using temperature modulated differential scanning calorimetry (DSC), which can separate total heat flow generated at the curing of a composite material into the non-reversing heat flow (exothermic component) and the reversing heat flow (specific heat component). The non-reversing heat flow component was used to determine the cure kinetics and the reversing heat flow was used to obtain the specific heat. The thermal conductivity was measured using periodic heating radiation thermometry, which can acquire physical properties not only in the thickness direction, but also in the in-plane direction. The measurement object was a prepreg, which was a carbon fiber woven fabric impregnated with an epoxy resin.

3.1 Cure kinetics and specific heat

For deriving the cure kinetics and specific heat of the composite material, a temperature modulated DSC was used. The conventional DSC outputs only the total heat flow including the

exothermic component and the specific heat component, but the temperature modulated DSC can extract the heat flow responding to the periodic temperature modulation (specific heat component) from the total heat flow by overlapping the sinusoidal wave temperature modulation of about +/-1°C to the constant speed temperature increase obtained by the ordinary DSC. In addition, by subtracting the specific heat component from the total heat flow, the exothermic component can be extracted. This method can accurately detect the specific heat component and the exothermic component of a composite material, so the improvement of the analysis accuracy can be expected.

Figure 3 and **Figure 4** present the cure kinetics and the specific heat measured at ramp rates of 1.0, 1.7, 3.5, and 6.0°C/min. The total heat flow can be separated into the exothermic component and the specific heat component, which can be applied to the analysis.

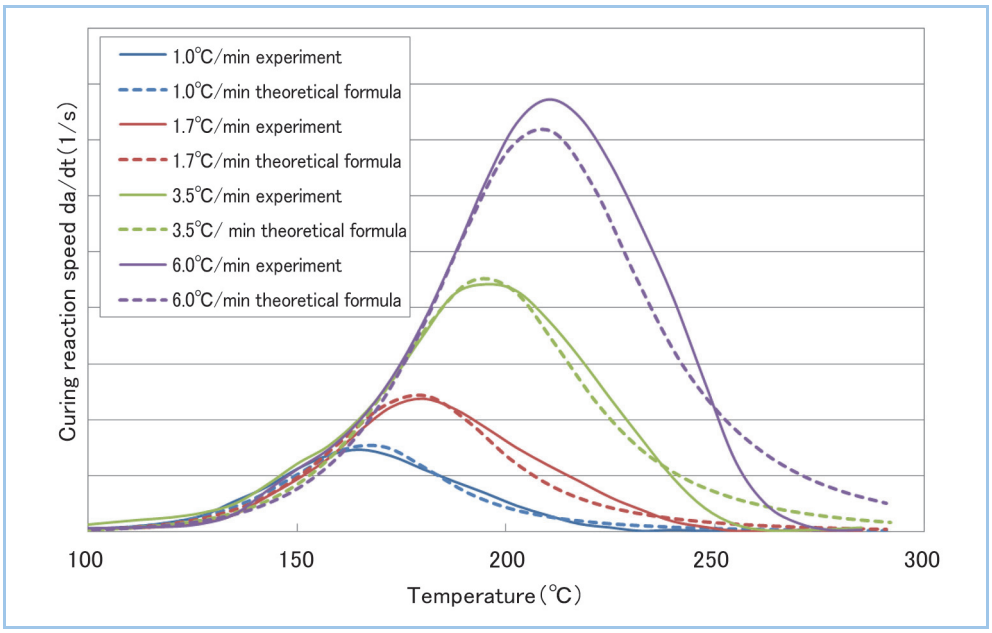


Figure 3 Derivation of cure kinetics
The results of fitting the parameters of the theoretical formula of the cure kinetics to the curing reaction speed (broken line) obtained by temperature modulation DSC are shown (solid line). Fitting was performed based on the nominal ramp rate of 1.7°C/min.

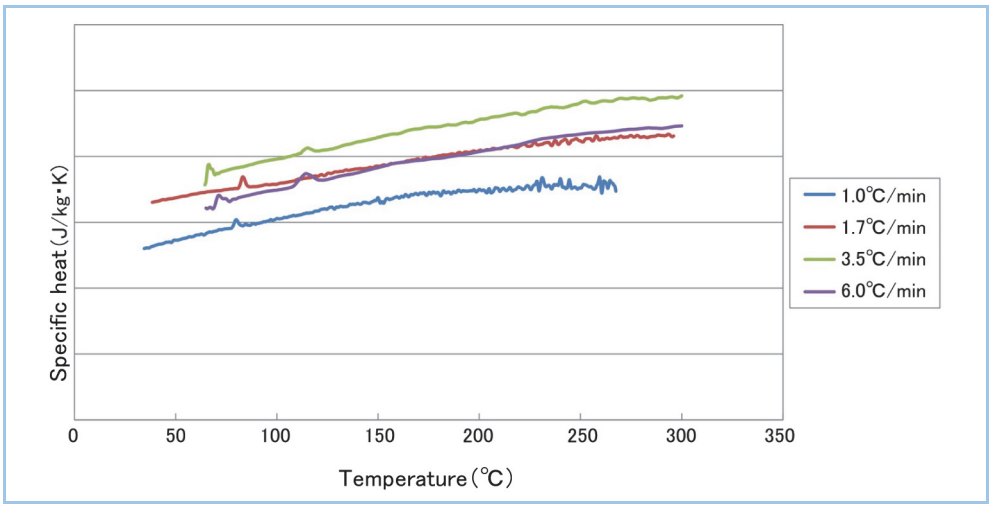


Figure 4 Measurement results of specific heat
The measurement results of specific heat at each ramp rate obtained by temperature modulation DSC are shown. Since the specific heat showed temperature dependence, it was applied to the analysis as a function of temperature.

3.2 Thermal conductivity

For the measurement of thermal conductivity, periodic heating radiation thermometry was used. A laser flash method is generally used as the measurement method of thermal conductivity, but its measurement direction is limited to the thickness direction of the test piece, so it is not suitable for the measurement of a composite material with thermal anisotropy. Therefore, a periodic

heating radiometric method was used, which can measure thermal conductivity not only in the thickness direction of the test piece, but also in the in-plane direction by periodically heating only the test piece and moving the radiation temperature measurement position.

Figure 5 shows the measurement results of thermal conductivity. The thermal conductivity in the in-plane direction (X) was higher in comparison with that in the thickness direction (Z), which indicated temperature dependency. Analysis in consideration of thermal anisotropy and temperature dependence of the composite material can be made.

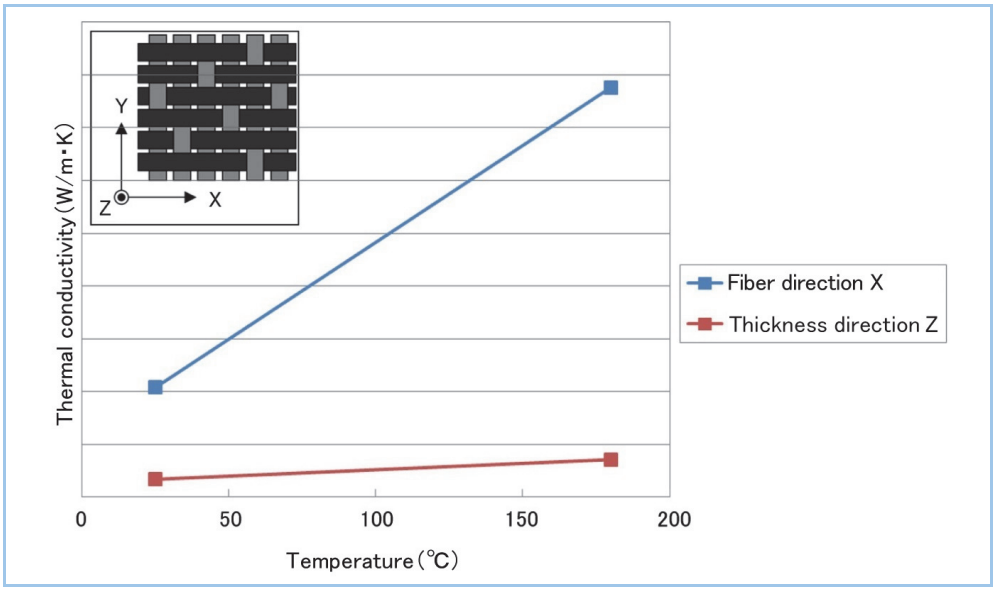


Figure 5 Measurement results of thermal conductivity
The measurement results of thermal conductivity by periodic heating radiometric method are shown. Since the analysis was carried out in two dimensions, physical properties in the X and Z directions were acquired. As the thermal conductivity showed temperature dependence, it was input to the analysis as a function of temperature.

4. Verification of analysis accuracy

4.1 Temperature measurement test of thick composite parts

To confirm the validity of the material properties measured in chapter 3, the internal temperature of a thick composite part during autoclave curing was measured and compared with the analysis results. As shown in **Figure 6**, thermocouples were installed at 3 points placed in the thickness direction (tool side, central part, and bag side) of the laminate sheet with a thickness of about 18 mm (64 ply × 0.29 mm/ply) to measure the temperature during curing. The size of the test piece was 300 mm × 300 mm.

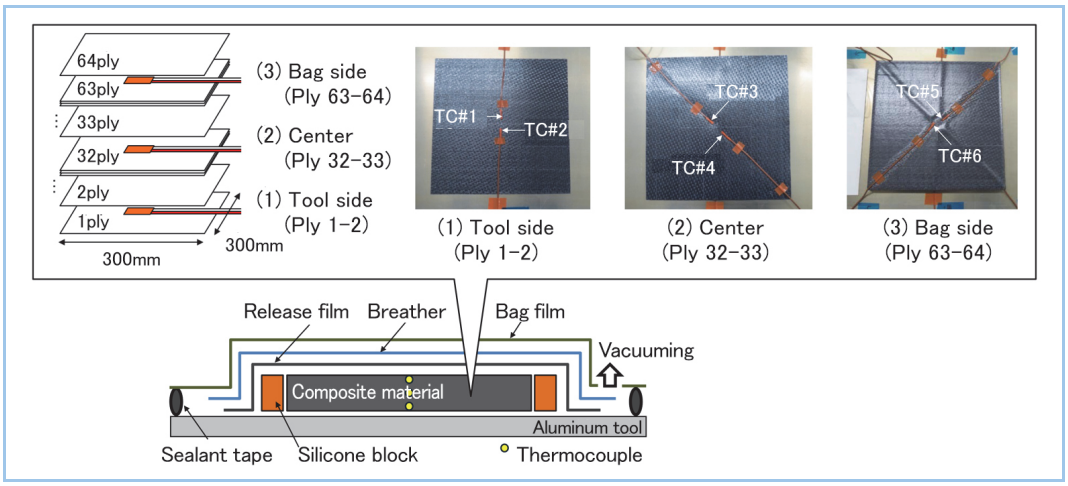


Figure 6 Bag configuration schematic diagram of verification test
A cross-sectional view of the bag configuration in the verification test is shown. Two thermocouples were installed at each of 3 points placed in the thickness direction of the composite material, and the temperature of the composite part during curing was measured.

A composite part was placed on an aluminum tool with a thickness of 10 mm, and a release film, a breather and a bag film were applied to the top of the composite. The composite material was insulated by placing silicone blocks around it to shut off heat input from the periphery. The curing conditions of the autoclave were set to a ramp rate of 1.7°C/min, a holding temperature of 185°C, a holding time of 120 minutes, and an autoclave pressure of 0.65 MPa.

4.2 Comparison of analysis and experiment

Figure 7 shows the curing analysis model. The analysis model was a two-dimensional cross-sectional model, to which the thermal properties measured in chapter 3 were input. The heat transfer coefficient with respect to the inside of the autoclave was set on the upper part of the composite material and the lower part of the tool, and the boundary conditions at the right and left ends of the composite material were adiabatic.

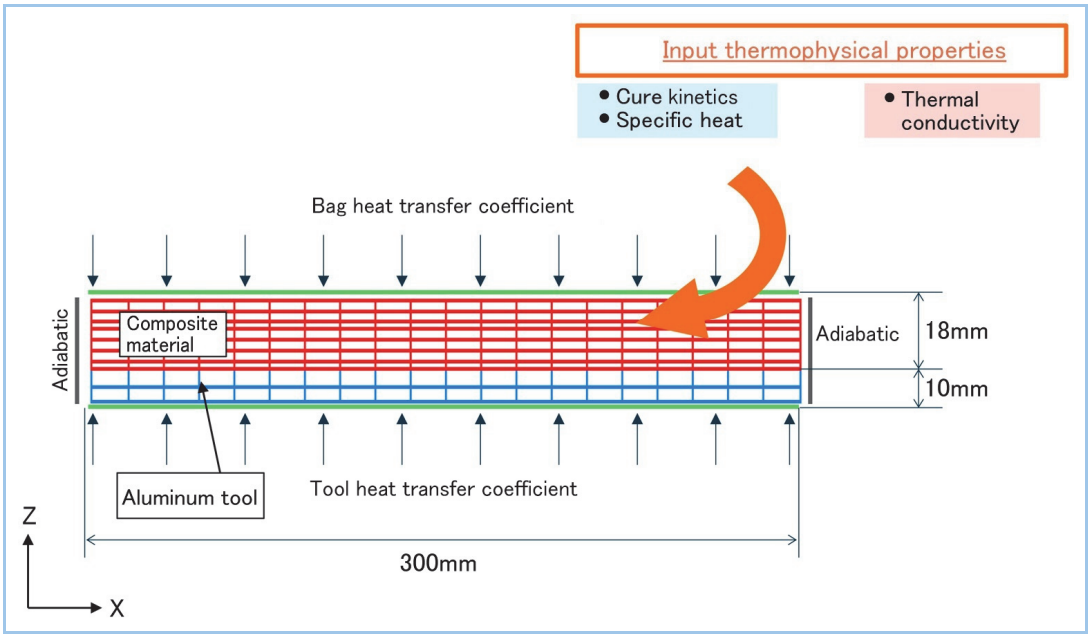


Figure 7 Curing analysis model
The model diagram used for curing analysis is shown. The configuration with the same conditions as the bag configuration in the verification test shown in Figure 6 is set. In the experiment, the width of the aluminum tool is larger than that of the composite material, but it is set to the same width due to the limitations of the functionality of the analysis software.

Figure 8 compares the temperature of 3 locations of the composite material in the thickness direction. For all 3 of the measured points placed in the thickness direction, the prediction accuracy was as follows: the difference in the curing heat generation peak temperature between the analysis results and the experimental results was 5°C or less, and the difference in the arrival time to the peak temperature was 3 minutes or less. The temperature decrease after the curing heat generation peak in the analysis results was gentler in comparison with that in the experiment results. This is presumably because the experiment used an aluminum tool larger than the composite part, but the analysis modeled an aluminum tool with the same width as the composite material in two dimensions. We will further improve the analysis accuracy by improving the modeling in the future.

As the above results of the verification test indicate, the analysis can predict the exothermic peak temperature and the peak temperature arrival time from the experiment, so the prospect of reducing the curing conditions setting cost through the application of the analysis has been achieved.

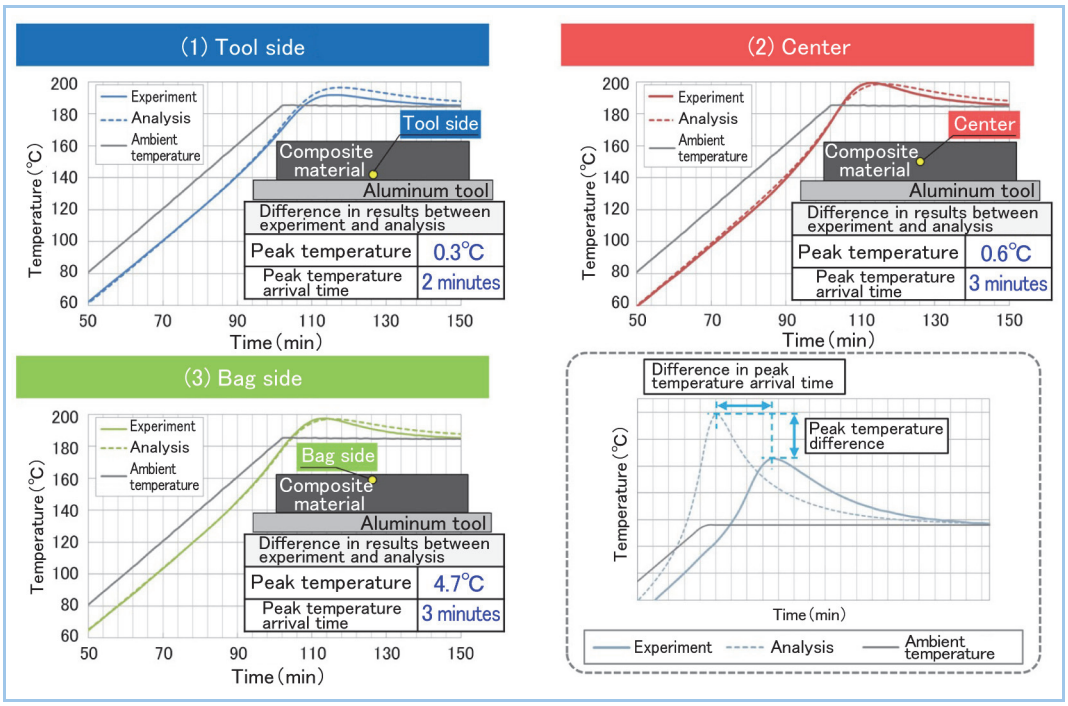


Figure 8 Analysis accuracy verification results
The results of the analysis accuracy verification test are shown. As a result of comparing the experiment (solid line) with the analysis (broken line), the difference in peak temperature at each measurement point was 5°C or less and the difference in peak temperature arrival time was 3 minutes or less.

5. Conclusion

This paper reported that we worked on accurately acquiring the thermophysical properties of composite materials, compared actual measurement data of a curing test with the analysis, and found that the analysis can predict experimental heat generation behavior with practically applicable accuracy. In the future, we will further improve the analysis accuracy.

Based on this result, the reduction of the number of part temperature measurement tests carried out in the curing conditions setting of composite parts is expected, and the prospect of reducing the curing conditions setting cost of aircraft parts by about 60% has been achieved. In the future, we will promote the practical application of this result and contribute to the improvement of the curing conditions setting process of composite material products.

References

(1) Noma, K., et al., High Accuracy Prediction of Temperature Profiles in Composite Parts During Autoclave Curing, 18th European Conference on Composite Materials, (2018)
(2) Convergent Manufacturing Technologies, Inc., https:www.convergent.ca/