EVALUATION AND VERIFICATION OF TEMPERATURE DEPENDENT OUT-OF-PLANE SHEAR MODULUS OF DISCONTINUOUS CARBON FIBER REINFORCED THERMOPLASTIC COMPOSITE BY MODIFIED DOUBLE NOTCHED COMPRESSION METHOD

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Keywords: CFRTP, out-of-plane shear modulus, temperature dependence, double notched shear test.

Abstract
This study focused on the temperature dependence of the out-of-plane shear modulus of discontinuous carbon fiber reinforced thermoplastic (CFRTP). And it verified the theoretical model based on viscoelasic behavior of matrix resin by using the novel test method. The new test method for out-of-plane shear properties of discontinuous CFRTP was suggested which was constructed based on "Standerd Test Method for In-plane Shear Strength of Reinforced Plastics" (ASTM D 3846-79). Accordeing to forming the optimum shape, it can be possible to measure the out-of-plane shear modulus G_{13} accurately by standard strain gauge. The temperature dependence of G_{13} for CFRTP was clarified experimentally by the proposed test method at various temperature conditions. And the validation of the theoretical model of G_{13}(T) with function of temperature was discussed by comparing with experimental results. As a result, the measurement G_{13} significantly agree with theoretical value G_{13}(T). So, the predictional model and the test method for out-of-plane shear behavior can be very useful to design the composite materials suitable for its products and predict the temperature influence.

1. Introduction

A weight reduction of transportation equipment has been important mission, since it leads to improve energy efficiency and reduce CO_2 emission and environmental load [1]. It is said that 75% of fuel consumption is directly occupied by vehicle weight [2]. So carbon fiber reinforced plastic (CFRP) has been recognized as a valuable material for a light-weight structure compared with steel. Therefore CFRP is widely expanding some applications in structure of aircrafts and automobiles. For example, discontinuous carbon fiber reinforced thermoplastics (CFRTP) is easy to be molded into several complex shapes with low manufacturing costs due to its high formability and make the structural design flexibility like a automobile structures so high [3,4]. From these situations, the quite in-plane isotropic

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CFRTP using polypropylene (PP)-based discontinuous CF-mat reinforced thermoplastics (CMT) has been developed (Fig.1). CMT has isotropic mechanical properties in-plane direction, not out-of-plane direction because of its fiber orientation [5,6]. So it causes so much unbalance between in-plane elastic modulus E_1 and out-of-plane shear modulus G_{13}, these mechanical parameters dominantly affect the flexural stiffness [7,8]. The authors also suggested that a prediction model of the contribution of G_{13} as a function of matrix resin particuraly influence on the much reduction of flexural modulus at high temperature condition compared with lower temperature theoretically [8]. However the theoretical $G_{13}(T)$ has been not completely verified by experimental procedure yet. Even though there are some methods to evaluate out-of-plane properties [9-12], measureing a experimental parameter of G_{13} is difficult because of occurring the un-uniformly strain distribution on each methods. Thus it is exactly faced with needs to develop a proper measurement method for accuracy G_{13}. So several researcher suggested some methods to estimate G_{13} [13,14]. For example, Bouette et al. used a single-lap shear specimen continuous CF and epoxy composite in order to estimate strain rate dependence of out-of-plane properties. They concidered the relationships between a shape of specimen and the strain distribution by using FEM analysis in order to find out the optimum specimen-design for preventing from misunderstanding some properties due to stress concentration and un-uniformly strain distribution. And they resulted that the dependent on strain-rate of G_{13} is not so much remarkable, in the case of unidirectional CF/epoxy composite material. On the other hand, the authors indicated that the influence of temperature change on G_{13} of CMT is much higher than strain rate dependence in previous study [8]. These results indicated that the viscelastic behavior of discontinuous CFRTP is considerably sensitive to temperature change, compared with strain rate change at the experimental condition.
Thus, this study focused on the temperature dependence of G_{13}, and verified the theoretical model based on the viscoelastic behavior of matrix resin by conducting the novel test method. And the test method proposed by the authors is based on “Standard Test Method for In-plane Shear Strength of Reinforced Plastics” of ASTM as Bouette suggested [11]. Improving the test method is to develop the overlap depth of the alternative shape of double notches (Fig.2) so that the shearing deformation area is expanded. The authors have anlized the optimum shape for causing and detecting the uniformly shear strain distribution by FEM model before measuring G_{13} the experimentally [15]. And, as a result, an optimum overlap depth of two notches was suggested in a case of not-so-thick specimen of discontinuous CFRTP in which chopped carbon fibers tapes were not oriented out-of-plane direction.
In this study, the newly testing method will be applied at several environmental temperatures in order to verify the validation of theoretical model for temperature dependence of G_{13}. Additionaly it is discussed the validation of the theoretical model for $G_{13}(T)$ including the matrix resin viscelastic parameter by comparing with the measurement results which is obtained by the proposed method.

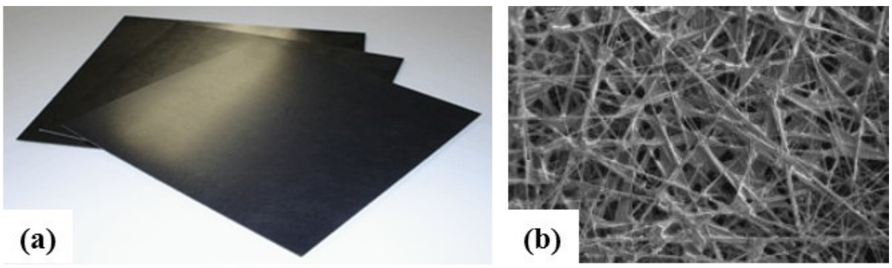

Fig.1 CMT perform (a) and carbon fiber mat (b).

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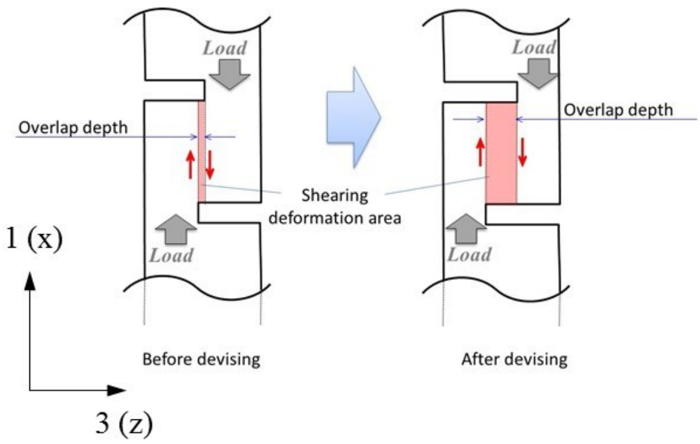

Fig.2 Conventional and proposed shear test method, comparing shear deformation area.

2. Constructing the model for the temperature dependence of out-of-plane shear modulus

Generally, the out-of-plane shear modulus of the continuous CFRP is derived from only using carbon fiber shear modulus G_f, volume fraction of fiber V_f and matrix resin shear modulus G_m, by using slab model [16]. The theoretical out-of-plane shear modulus of CMT is also obtained similar model by referring the slab model which was modified to laminated both the in-plane isotropic CF-mat layer and matrix resin layer as shown in Fig.3. So, considering the shape coefficient value α unique to the dispersed and randomly-oriented carbon-fiber mat, theoretical model G_{13}(T) is represented into the following equation for discontinuous CFRTP [8].

$$G_{13}(T) = \frac{\alpha \cdot G_f G_m(T)}{V_f G_m(T) + \alpha(1 - V_f)G_f} \tag{1}$$

Here, α means the parameter of fiber reinforcement utilization dependent on the average of fiber lengths and the homogeneousness of fiber orientations. And the parameter α assumes constant at various temperature condition. The value α of the CMT was validated in author's previous study [8]. G_f is defined as 13.7 GPa referring to the experimentally measured value by Fujita et al. [17]. Then G_m(T) is matrix resin shear modulus as a function of temperature T. It is introduced by using the Young's modulus of matrix resin E_m(T) as the following.

$$G_m(T) = \frac{E_m(T)}{2(1+\nu)} \tag{2}$$

Hereν is Poisson's ratio and E_m(T) is the Young's modulus of the resin as a function of temperature T, considering its viscoelastic property. The viscoelastic properties are obtained as storage modulus E_m'(T) which has temperature dependence by dynamic mechanical analysis (DMA) as followed equation:

$$E_m(T) = E_m{}'(T) \cdot \{(\tan\delta)^2 + 1\}$$

$$* \tan\delta = \frac{1}{\omega\tau} \tag{3}$$

Where, the loss tangent (*tanδ*) represents the phase difference between the strain-input and stress-output, and includes the frequency ω and relaxation time τ as a time function. From equation (1), (2) and (3), $G_{13}(T)$ as function of temperature can be predicted by only measuring viscoelastic properties of matrix resin.

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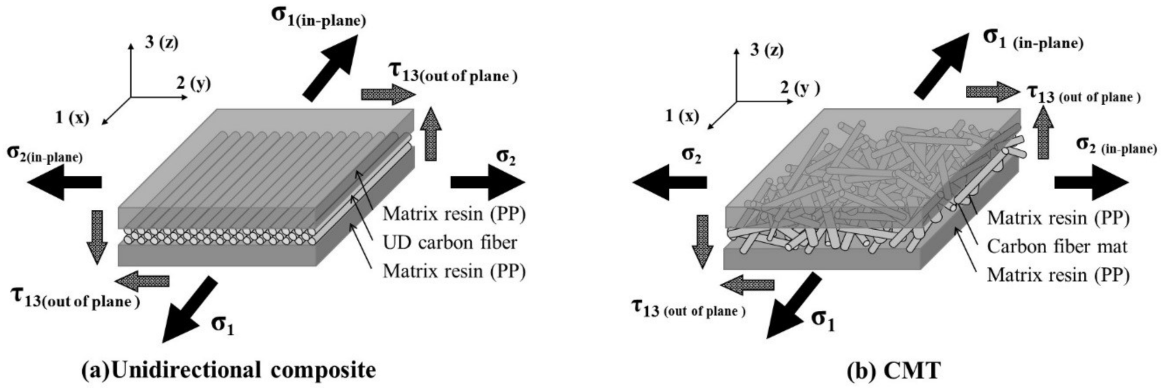

Fig.3 Slab models for continuous and discontinuous composite laminates.

3. Experimental procedure
3.1. Preparation of specimen

The CMT preform sheets used as an intermediate material consisted of a discontinuous CF-mat impregnated with polypropylene (PP) as matrix resin. The CF mat was producted by TORAY industries, Inc. with T700 (tensile modulus; 230 GPa, tensile strength; 4900 MPa) chopped and dispersed in the state of mono-filaments. In this study, the length of chopped CF was controlled of around 6.0 mm. The impregnated CMT preform sheets were manufactured through hot-melt process by using continuous press machine.
The specimens of CMT plate with the thickness about 4.5 mm were obtained from laminated 8 CMT preform sheets by the compression molding process. The laminations were set on mold, heated up at 220 degree Celsius for 2 minutes without pressure, after that, were kept at pressure 5 MPa for 2 minutes by press machine. And then, the laminates were cooled down at the same pressure for 1 minute, and the in-plane isotropic CMT plates were obtained. The fiber volume fraction V_f of CMT plates was 20%.

3.2. Designing shape of specimen

The specimen for measuring G_{13} was designed based on ASTM D3846-79 [11], as a double notched shear specimen. There are some devised points on the shape to measure the accurate out-of-plane shear strain. For example, increasing the overlap depth between notches makes the region of uniform shear strain distribution expand. However the excess of overlap depth causes a compression buckling at the bottom of notches by compressive load without a shear deformation in the gauge section. It is needed to decide the optimum overlap depth for measuring shear modulus by using a strain gauge. The appropriate shape of the specimen was determined by using FEM model in the author's previous research [15].
The DNC-S specimens were cut from CMT plates and attached to tabs before shaping the notches in order to prevent from buckling at the bottom of notches. As Fig.4 shows, the dimension of specimen is 80mm of total length: l, 15mm of width: w, and 9.5 mm of the total thickness added two tabs: t. Then the distance of both notches: d is 6.4mm, the depth of notches is t/2+0.5 mm, in short, the overlap depth: a is 1.0 mm.

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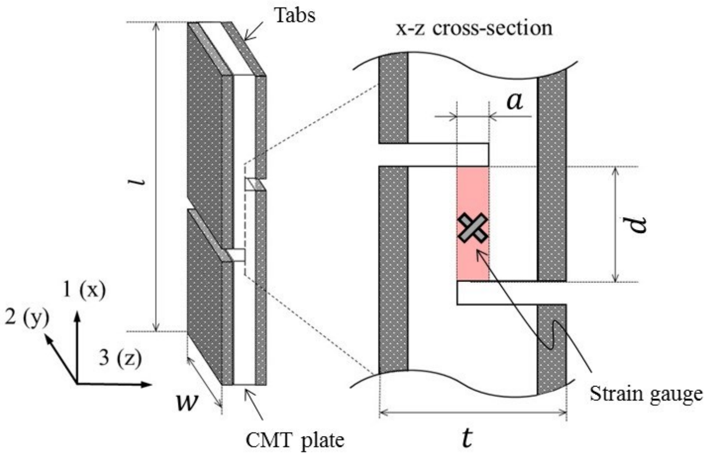

Fig. 4 Dimensions of newly DNC-S test specimen.

3.3. Dynamic viscoelasticity of PP matrix [8]

Temperature dependence of the viscoelastic properties (storage modulus: Em' and loss tangent: tanδ) of PP was evaluated by dynamic mechanical analysis (DMA) using a viscoelastometer instrument (TA instruments, RSA-III) in dual cantilever bending mode. These test were conducted with an applied strain sine wave amplitude of 0.01%, dynamic frequency of 1.0 Hz and environmental temperature ranging from -30 degree Celsius to 150 degree Celsius with interval of 5 degree Celsius.

3.4. Evaluation the out-of-plane shear strains and shear stress

The out-of-plane shear strains were evaluated by compression test at 3 kinds of temperature conditions of -30, 25, 75 degree Celsius in the thermostat chmber by using a precision universal testing machine (AUTOGRAPH AG-X Plus: manufactured by Shimadzu Corporation). The compression loads applied to specimens with crosshead speed of 0.5 mm/min. The shear strains were detected by ±45˚strain gauges toward compression loading direction (Fig.4). The engineering shear strain was given as the difference of the measured two strains. And the shear stress was calculated from the compression load divided by the shearing area, i.e. *d*: 6.4 mm x *w*:15 mm.

3.5. Evaluation the out-of-plane shear modulus

G_{13} was calcurated from the relationships between shear stress and shear strain. Since purpose of the experiment was to obtain the shear modulus, so the experimental value were recorded until 2.0 % shear strain enough to calcurate the shear modulus. And G_{13} was evaluated at liner region on the shear stress-strain curves.

4. Results and discussions
4.1. Results of DNC-S test at various temperature

Fig. 5 shows the out-of-plane shear stress and strain curve obtained by the DNC-S test method at 3 kinds of temperatures. Each results exactly have liner slope at extremely initial strain region, so it can be evaluated the G_{13}. And the slope of liner region increased with lower temperature condition. According to these results, the temperature dependence of G_{13} exactly became clear by conducting the DNC-S test at some temperatures.

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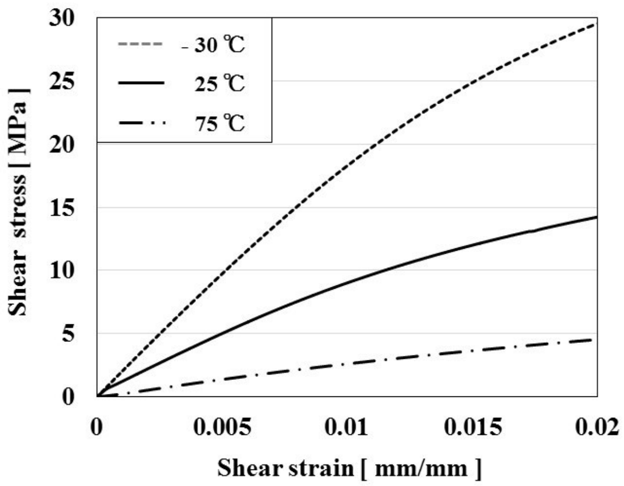

Fig. 5 Stress-strain curves from DNC-S test at specified temperatures.

4.2. Temperature dependence of the matrix resin viscoelastic properties

In order to consider the effect of temperature dependence of matrix resin on G_{13}, the temperature dependence of Young's modulus E_m(T) was measured by using DMA test and the equation (3). The result, shown in Fig. 6, obviously shows the temperature dependence of applied PP. As a result, it is clear that G_m required to calcurate the theoretical value $G_{13}(T)$ also has the temperature dependnence by reffering the equation (2).

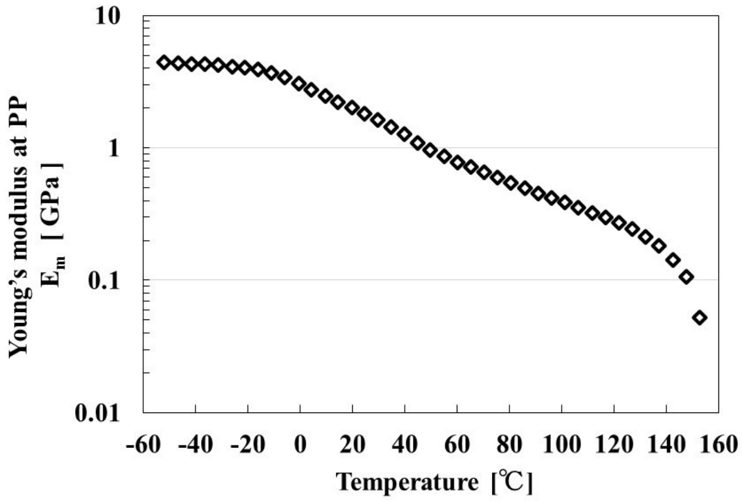

Fig. 6 Young's modulus of PP at various temperature.

4.3. Considering the velification of the theoretical model for out-of-plane shear modulus

This paragraph verifies the proposed theoretical model for G_{13}(T) by comparing with measured G_{13} experimantally. The theoretical predicted value G_{13}(T) include a function of temperature of matrix resin is calcurated from equation (1), in this case, some parameters excepted G_m(T) of the Poison's ratio ν, the shape coefficient value α and the CF shear modulus G_f are applied an already-known value which was validated in the author's previous paper (Table 1). These parameter were used as hypothesized value for the propsed model on the previous paper [8], and the values have been also demonstrated by verifying the validity of the proposed model. And G_m(T) is obtained from both the equation (2) and (3) by measuring E_m(T). And then theoretical temperature dependentce of G_{13}(T) is represented as a real line on Fig.7. This result also indicates that theoretical out-of-plane shear modulus of CMT obviously has temperature dependency at range of experimental temperature. Additionaly, the experimental obtained

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values G_{13} from the proposed shear test method are indicated as symbols on the same graph. Comparing the theoretical line with the experimentally-obtained values, they are certainly good agreement with both the tendency and the value of G_{13} at 3 kinds of temperature. The experimentally demonstration by DNC-S test confirmed the validation of the proposed theoretical model for temperature dependent on out-of-plane modulus of CMT. And the DNC-S test method also be utilized for measuring the out-of-plane shear modulus with various temperature conditions.

Table 1 Defined parameter for theoretical equations [8].

Temperature [℃]	Poisson's ratio: ν [-]	shear modulus of CF: G_f [GPa]	shape coefficient value: α [-]
-30	0.30		
25	0.40	13.7	0.279
75	0.50		

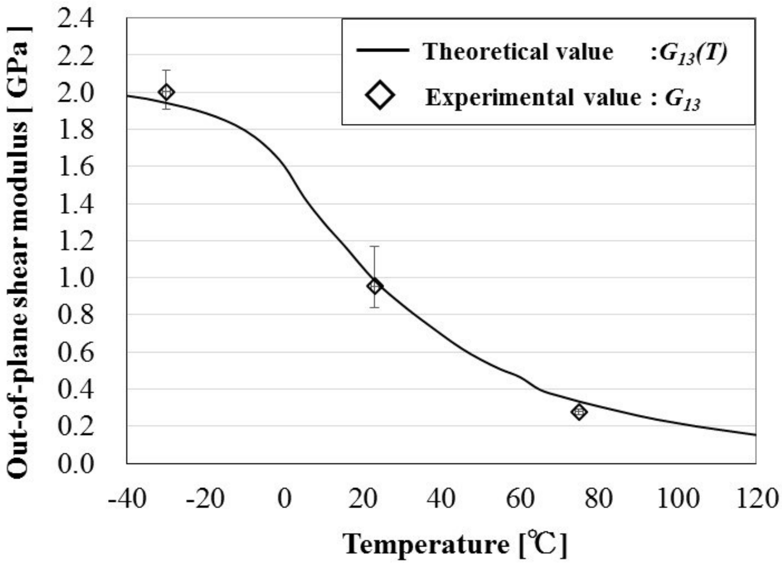

Fig. 7 Comparison of experimental G_{13} and theoretical G_{13}(T) on various temperature.

5. Conclusion

This study focused on the temperature dependence of the out-of-plane shear modulus of discontinuous CFRTP, which consist of in-plane isotropic CF mat and PP as matrix resin. The modified shear test based on ASTM standard method of DNC was proposed and experimentally verified, in order to obtain the G_{13} accurately. Thus the proposed test method can evaluate the shear properties at various temperature, so it means the DNC-S test is useful method to detect G_{13} accuratly at various environment. And then the validation of theoretical model for G_{13} was also verified, so it indicate that the G_{13} of CMT was strongly reflected by the viscoelastic property of matrix resin. As a result, it became clear that G_{13}, which was conventionally difficult to be detected directly, can be predicted easily and accurately by DMA test results of matrix resin and some predetermined parameters such as the reinforcement coefficient, the fiber volume fraction and the shear modulus of carbon fiber. So, the theoretical model and the test method for the out-of-plane shear behavior can be useful to design for some products and improving the technology of safty design by accurately prediction.

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References

[1] J. Takahashi & T. Ishikawa: “Recent Japanese activity in CFRTP for mass production automobile” *Proceedings of ITHEC 2014.* Bremen, (2014-10), pp. 30-31.

[2] K. Friedrich, A. A. Almajid: “Manufacturing aspects of advanced polymer composites for automotive applications”, *Applied Composite Material*, (2013), 20, 107-128.

[3] J. Takahashi, T. Ishikawa: Next challenge in CFRTP for mass production automotive application, *Proceedings of SAMPE SEICO14* (2014), Paris, France, pp.181-188.

[4] F. Rezaei, R. Yunus , N. A. Ibrahim, E. S. Mahdi.: Development of short-carbon-fiber reinforced polypropylene composite for car bonnet, *Polymer-Plastics Technology and Engineering*, 47:4 (2008), 351-357.

[5] N. Hirano, H. Muramatsu, T. Inoue: Study of fiber length and fiber-mtrix adhesion in carbon-fiber-reinforced polypropylenes, *Adv. Compos. Mater.,* 23, 2,(2014), 151-161.

[6] M. Hashimoto, T. Okabe, T. Sasayama, H, Matsutani and M. Nishikawa.: Prediction of tensile strength of discontinuous fiber/polypropylene composite with fiber orientation distribution. *Compos part A* 43 (2012), 1791-1799.

[7] G. Caprino, P, Iaccarino, A. Lamboglia: The effect of shear on the rigidity in three-point bending of unidirectional CFRP laminates made of T800H/3900-2, *Compos. Struct.* 88 (2009), 360-366.

[8] W. Nagatsuka, T. Matsuo, F. Yano, K. Furukawa and J. Takahashi: Formulation about time- and temperature-dependent flexural modulus of discontinuous carbon fiber mat reinforced thermoplastics, *Proceedings of 20th International Conference of Composite Materials*, Copenhagen, (2015-7), https://s3-eu-west-1.amazonaws.com/iccm20/iccm20-v2.zip.

[9] ASTM D2344; Apparent inter-laminar shear strength of parallel fiber composites by short-beam method. *American Society for Testing and Materials* (1984).

[10] ASTM D5379-12; Standard Test Method for Shear Properties of Composite Materials by V-Notched Beam Method. *American Society for Testing and Materials* (1993).

[11] ASTM D 3846-79; Standard Test Method for In-plane Shear Strength of Reinforced Plastics, *American Society for Testing and Materials* (1985).

[12] P. Morgan: *Carbon Fiber and their Composites,* (2005), pp.699-705.

[13] B. Bouette, C. Cazeneuve and C. Oytana: Effect of strain rate on interlaminar shear properties of carbon/epoxy composites, Compos. Sci. Technol. 45 (1992), 313-321.

[14] S.R. Hallett, C. Ruiz, J. Harding.: The effect of strain rate on the inerlaminar shear strength of a carbon/epoxy cross-ply laminate: comparison between experimental and numerical prediction. *Compos. Sci. Technol.* 59, (1999), 749-758.

[15] T. Matsuo, T. Murakami, M. Kan, T. Sumiyama and K. Sakaguchi.: New test method for nonlinear out-of-plane shear property discontinuous and randomly-oriented hermoplastic CFRP. *The proceedings of the symposium on composite materials,* Kanazawa, (2015).

[16] D. Hull, T.W. Clyne, An Introduction to Composite Materials (2nd Ed), Cambridge Solid State Science Series, *Cambridge University Press* (1996), pp.60-77.

[17] K. Fujita, M. Kojima and T. Iwashita. *Material Testing Research Association of Japan.* 58, 3, (2013), 143-148.

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