Measurement of elastic constants of a structural adhesive using pulse-echo method

超音波を用いた構造用接着剤の弾性率の測定

Shinobu Sugasawa^{1†}, Takahiro Ando², Shigeru Akiyama³, and Toshiaki Iwata⁴ (¹⁴National Maritime Research Institute) 菅澤 忍^{1†}, 安藤 孝弘², 秋山 繁³, 岩田 知明⁴ (¹⁴(独) 海上技術安全研究所)

1. Introduction

Adhesives classified by the term "structural adhesive" are characterized generally to ones having high reliability and holding low degradation of adhesion properties e.g. adhesive strength and environmental resistance under a large load for a long time. Structural adhesive is used widely for aviation, manufacturing industry such as automotive, building construction and ship building. The composition of many commercial adhesives is homogeneous, but that of structural adhesive is the composite of e.g. resin and elastomer in order to provide excellent adhesive characteristics to adhesiveness. In the field of ultrasonics, there are many researches related to composite materials of resin and fiber so-called fiber reinforced plastics. On the other hand, it seems that the study of adhesive has interest in not compositional but material. Therefore, homogeneous structural adhesive formed by blending several materials will be worth studying by ultrasonic technique.

In this study, a structural adhesive composed of a resin composed of PMMA (=Poly(methyl methacrylate) dispersed elastomer particles is investigated by pulse-echo method, and the sound velocity and the elastic constants of the adhesive are evaluated, and finally the Young's modulus derived from the sound velocity is compared with that measured by static tensile test.

2. Experimental Method

An adhesive used as specimen was a structural adhesive manufactured by ITW PP&F Japan Co., Ltd., which was two-liquid blending type, and was composed of PMMA dispersed elastomer particles, but the ratio of resin to elastomer and the physical properties of the elastomer were unknown.

Specimen for ultrasonic measurement was prepared as follows: Adhesive was curing on an aluminum plate of 10 mm-thickness using a mold, and a plateau of $15 \times 15 \text{ mm}^2 \times 5 \text{ mm-thickness}$ was formed on this plate after curing. The surface of the plateau was polished so as to be smooth and parallel to that of the plate. Specimen for tensile test was prepared as conforming to JIS K7162 1B. The

speed of the tensile test was 0.5 mm/min while the displacement of the specimen was below 3 mm. The density of the adhesive was estimated by Archimedes method and was 1.097 g/cm^3 .

The center frequencies of both longitudinal transverse ultrasonic sensors used in and measurement were 6 and 4 MHz, respectively. Ultrasonic pulse was introduced from the surface opposite to the adhesive on the plate; the echoes of the pulse were observed on a digital oscilloscope (Fig.1). Since only echoes of the plate appeared, the specimen was polished further. When the thickness of the adhesive became about 1.5 mm, longitudinal wave echoes appeared in the adhesive. Furthermore, both longitudinal and transverse wave echoes in the adhesive became distinctly visible after the thickness decreased to about 0.5 mm. Ultrasonic measurement was done when the thickness had reached 0.520 mm. Elastic constants were calculated by the formula by which these values of a material can be derived from the density and sound velocities of longitudinal and transverse waves of the material. $^{1)}$



Fig.1 Measurement system

3. Results and Discussion

Longitudinal and transverse wave forms observed in the experiment are shown in **Fig. 2**. The velocities of these wave motions were calculated from the time difference between "S" and " e_1 " echoes and the thickness of the adhesive. **Table I** shows elastic constants using the formula mentioned above.¹⁾ In addition to this, the attenuation coefficients of longitudinal and



Ultrasonic echoes obtained in experiment ("S" indicates reflection echo of the interface Fig. 2 between adhesive and aluminum plate and "e" does bottom echo of adhesive.)

Table I Results of analysis (long.=longitudinal, trans.=transverse)								
thickness (mm)	round-trip time		velocity		density	bulk	Young's	Poisson's
	long.	trans	long.	trans.	(kg/m^3)	modulus	iodulus modulus rati	ratio
	(µs)	(µs)	(m/s)	(m/s)		(GPa)	(GPa)	iullo
0.520	0.480	1.159	2,165	897.5	1,097	3.96	2.47	0.396

. . . .

transverse waves were 0.383 and 0.928 neper/mm, respectively. The Young's modulus of the specimen is 2.47 GPa and equal to the nominal value of PMMA to the same degree. On the other hand, Poisson's ratio is larger than the nominal value of PMMA. It is thought that elastomer contained in the adhesive causes this phenomenon, since the poisson's ratio of elastomer approximates to 0.5. Similarly, high absorption of wave energy in propagation will be explained by the existence of elastomer in the adhesive.

Fig. 3 shows the stress-strain curve obtained from a tensile test of the adhesive. From the analysis of this curve, Young's modulus and yield stress were estimated at 741 MPa and 12 MPa, respectively. Young's modulus was calculated by applying least-square method to the interval of stress between 0 - 3 MPa.

From the results of the analysis, it should be noted that there exists a remarkable difference of Young's modulus between the values obtained from



Fig. 3 Stress-strain curve of specimen

the ultrasonic measurement and the tensile test. This will be able to be explained as follows: The Young's modulus of elastomer exists generally between 1 MPa and 10 MPa under a static load. Therefore, if PMMA includes elastomer, it will be expected from the rule of mixtures in materials that the elastomer in the adhesive contributes little to the whole Young's modulus since Young's modulus of PMMA is about 3 GPa. On the other hand, if a load changes periodically and the frequency of this oscillation is to degree of Megaherts, the Young's modulus of elastomer is considered to increase to order of GPa. Because it is usually difficult to make ultrasonic measurement of elastomer, this expectation is seemed to be of interest to some extent.

Finally, although elastic constants can be deduced easily from ultrasonic measurement data by the formula¹ mentioned in **2.**, it seems that it is necessary to apply this formula carefully to case of composite material like structural adhesive we treated in this paper.

4. Summary

A structural adhesive was examined by both the methods of ultrasonic measurement and static tensile test. From the results, the elastic constants of the adhesive were estimated. It was found that there exists a great gap between Young's moduli obtained by both these methods. We attempted to explain this gap by the vibratory behavior of elastomer that was dispersed in resin.

References

1. Cho-onpa editorial committee: Cho-onpa Binran (Maruzen, Tokyo, 1999) [in Japanese] p.299.