

# Improvement of Impact Fatigue Strength of Adhesive Joint by CTBN Modification

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## Synopsis

Impact fatigue behaviors of the steel/CTBN-modified adhesive/steel butt joint were investigated. The adhesive butt joint specimens used in the present work were bonded with epoxy-polyamide and CTBN-modified epoxy-polyamide adhesives. Fatigue tests were also conducted under non-impact stress conditions to compare with the results from the impact fatigue test. The experiments showed that for the joint specimen from the adhesive modified with the CTBN the fatigue strength becomes higher under both of the stress conditions. In particular, the fatigue strength was improved remarkably under impact stress condition, that is, the distinct stress cycles dependence of impact strength was decreased by modifying the adhesive with CTBN. Furthermore, the effect of adhesive thickness on the fatigue strength was also discussed for the adhesive joint modified with CTBN. Under impact stress conditions, the relation between the fatigue strength and the adhesive layer thickness is different from that under the nonimpact one.

## INTRODUCTION

Adhesive bonding has attracted special interest recently as a joining technique of mechanical structure, for it offers potential advantages of saving the material costs and of simplified manufacturing processes. Therefore, mechanical properties of adhesive joints have come up as a serious problem, and many studies on this problem have been mainly conducted under static stress conditions.

From a view point of adhesive joints as mechanical structures, fatigue behaviors under service load conditions are of significance, but such investigations are few.<sup>1</sup> In general, impact fatigue load conditions are considered to be more severe among several service load conditions, and for several metallic materials it is well known that peculiar phenomena are observed under such a condition.<sup>2</sup>

The authors have performed a series of studies on the impact fatigue behaviors of the adhesive-bonded butt joint specimens.<sup>3,4</sup> The main purpose of the present study is to improve the impact fatigue strength of an adhesive joint by adding carboxy-terminated butadiene acrylonitrile (CTBN) into the adhesive as an improving agent.<sup>5</sup> The contribution of CTBN and the effect of adhesive thickness on the impact fatigue strength were discussed in comparison with the results obtained under nonimpact stress conditions.

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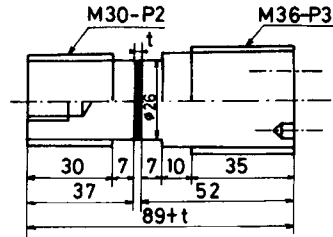


Fig. 1. Shape and sizes of adhesive-bonded butt joint specimen.

## EXPERIMENTAL

### Materials and Preparation of the Adhesive-Bonded Butt Joint Specimen

In this study, an epoxy resin (Epikote 828 from Shell Chemical Co., Ltd.) and a polyamide (Versamid 140 from Henkel Japan Co., Ltd.) were adopted as adhesive components; CTBN was used to improve the fatigue strength of the epoxy polymer. As an adherend material, a 0.15% C carbon steel conforming to JIS, S15C, was used. In case of the epoxy modification, an epoxy/CTBN ratio in weight was 1/1; the preparation was carried out without catalyst for 1.5 h at 170°C under stirred condition in an atmosphere of nitrogen.

The adherend material was machine-finished to obtain the shape and dimensions specified in Figure 1. The end face of the adherend specimen to be bonded was polished with an emery paper of the grade 320 mesh under dry condition. Surface roughness  $R_{\max}$  of the polished surface of the adherend specimen was about 1.6  $\mu\text{m}$ , where  $R_{\max}$  means the maximum height in the evaluation of surface roughness.

Two sorts of adhesive systems were chosen in the present study: the epoxy-polyamide system (epoxy/polyamide = 6/4 in weight) and the modified epoxy-polyamide system (modified epoxy/polyamide = 12/4 in weight).

An adhesive-bonded butt joint specimen was prepared as follows: A pair of the adherend specimen was polished; the adhesive joint specimen was cured for 2 h at 110°C and then cooled in a furnace. The butt joint specimen thus obtained was allowed to stand for 24 h at room temperature, and was subjected to a series of fatigue tests.

Adhesive layer thickness of the specimen was controlled by inserting a few glass beads of 0.1 mm or 0.5 mm in diameter between the two adherend end faces.

### Experimental Apparatus and Procedure

Principles of a push-pull fatigue testing machine and a tensile impact fatigue testing machine used in this work are illustrated in Figures 2 and 3, respectively. The full details are available elsewhere.<sup>3,4,6,7</sup> A brief explanation for the push-pull fatigue testing machine is given below. Fully reversed push-pull load acts on the specimen from the horizontal movement of a vibro-motor at about 3600 rpm. Load magnitude can be adjusted by changing the magnitude of eccentric masses attached to both ends of a rotor shaft.

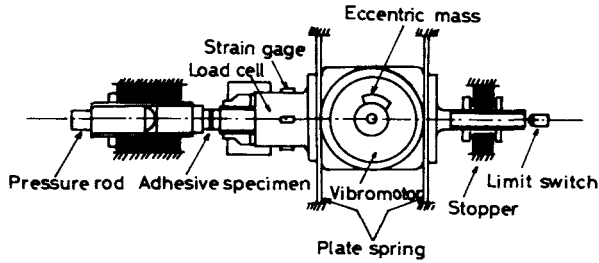


Fig. 2. Principle of the push-pull fatigue testing machine.

The tensile impact fatigue testing machine had been on trial manufactured by Chatani.<sup>8</sup> In Figure 3, the impact tensile stress caused from the collision between a steel pipe and stopper propagates along a steel rod to the test specimen fixed between a block and the end of the rod. The magnitude of impact load is adjusted by changing the distance of eccentricity of a crank as well as the rotating rate of the crank shaft in the range of 360–600 rpm using a steplless speed change device. The magnitude of impact load is monitored by means of the strain gage cemented on the steel rod near the test specimen. The details of the chuck device and load cell are given in Figure 4.

Typical results of the impact stress patterns obtained on both locations are shown in Figure 5. From the photographs, no significant difference in the impact stress patterns is observed between the steel rod and the test specimen. Also, the ratio of the maximum compressive stress  $\sigma_{cmax}$  to the maximum tensile stress  $\sigma_{tmax}$  (stress ratio  $R = \sigma_{cmax}/\sigma_{tmax}$ ) is found to be about  $-0.45$ .

In the same manner as shown in previous papers,<sup>3,4</sup> the output ratio of gage mounted on the test specimen to that on the rod was about 0.85. This value of 0.85 was used as a load transfer constant for the impact load of the testing machine.

Furthermore, both the rigidity and the loss tangent of bulk adhesives were determined on a torsional pendulum type viscoelastic tester (Resca Co., Ltd.); the tensile strength of bulk adhesive was measured on a universal testing machine (Autograph, Shimazu Co., Ltd.) based on the testing method designated according to ASTM, D-980-66. The tensile speed was 1 mm/min, and all the measurements were conducted at a constant ambient temperature of 20°C.

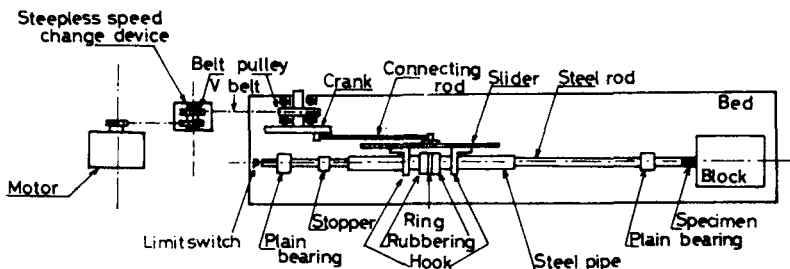


Fig. 3. Principle of the tensile impact fatigue testing machine.

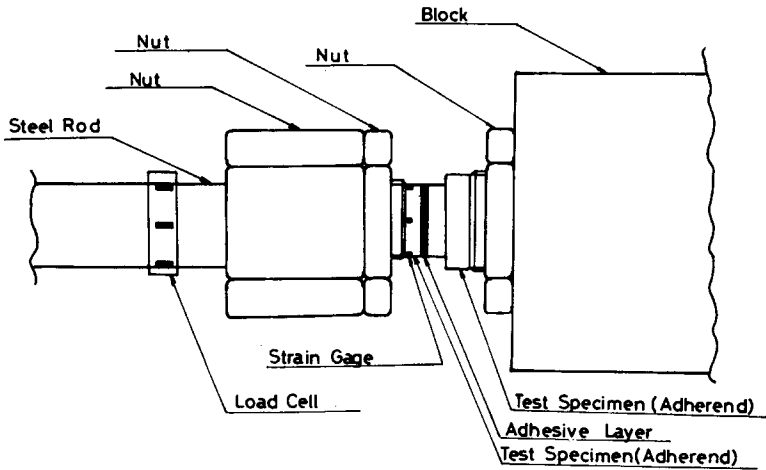


Fig. 4. Details of chuck device and load cell.

## RESULTS AND DISCUSSION

### Effect of CTBN Modification on the Fatigue Strength

Mechanical properties of three types of the bulk adhesives used in this work are given in Table I, together with the results of Epikote 828-Versamid 115 adhesive used in previous works.<sup>3,4</sup> The table shows that the Epikote 828-Versamid 115 material has the lowest rigidity. In addition, as the rigidity of adhesive increases, the loss tangent decreases, whereas the tensile strength increases. Such a behavior agrees with the general trend observed for polymer solids.<sup>9</sup> Besides, for the adhesive modified with CTBN the rubber particles are separated from the epoxy matrix.<sup>10,11</sup> The other adhesives, however, are composed of epoxy resin and polyamide resin; hence no precipitate was separated from the epoxy matrix.

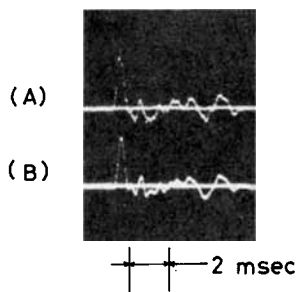


Fig. 5. Impact stress patterns. Stress waves detected from (A) load cell and (B) test specimen.

TABLE I  
Mechanical Properties of Bulk Adhesives

Adhesive	Tensile strength $\sigma$ (MPa)	Shear modulus $G$ (MPa)	Loss tangent $\tan \delta$
Epikote 828-Versamid 115	14.3	500	0.350
Epikote 828-Versamid 140	61.0	7810	0.023
Epikote 828-Versamid 140-CTBN	16.1	1580	0.073

Here, the influence of adhesive types on the fatigue strength was tested under nonimpact stress conditions of  $R = -1.0$  with the joint specimens whose adhesive thickness is 0.1 mm thick. Results obtained are shown in Figure 6 on an  $S-N$  diagram. The figure indicates that the fatigue strength increases from the CTBN modification and also the Epikote 828-Versamid 115 adhesive joint has the highest strength among the three adhesives.

Figure 7 shows the correlation between the loss tangent and endurance limit or the time dependent strength at  $10^7$  stress cycles. The figure suggests a good correlation between these properties.<sup>9</sup> In general, toughness of adhesive joints as well as the impact strength is increased by CTBN modification of the adhesive.<sup>5,12</sup> One of the reasons for the increase of the fatigue strength would be due to the fact that the loss tangent of adhesive increases with the CTBN modification.

Figure 8 shows the results for the impact fatigue test, in which the maximum tensile stress  $\sigma_{t_{max}}$  is taken as the ordinate. Comparing the fatigue strength behaviors under impact and nonimpact loads in Figures 6 and 8, we may conclude as follows. The impact fatigue strength is higher than nonimpact fatigue in a relatively low stress cycles range consistent to the single impact tests.<sup>13,14</sup> As the number of stress cycles increases, however, the impact fatigue strength decreases more rapidly than the nonimpact one. Furthermore, as was found in the previous studies done using the adhesive joint of Epikote 828-Versamid 115,<sup>3,4</sup> the fatigue strength under impact stress conditions approaches that under nonimpact stress conditions of about  $2 \times 10^6$  of stress cycles. Also we have demonstrated<sup>3</sup> that the slope of the  $S-N$  curve under impact stress conditions

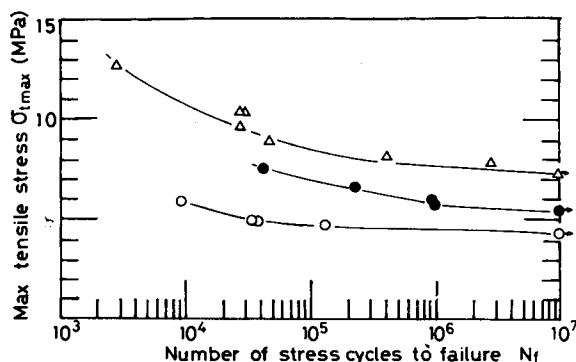


Fig. 6. Results of nonimpact fatigue tests (adhesive thickness  $t = 0.1$  mm): (●) Epikote 828-CTBN-Versamid 140; (○) Epikote 828-Versamid 140; (Δ) Epikote 828-Versamid 115.<sup>3,4</sup>

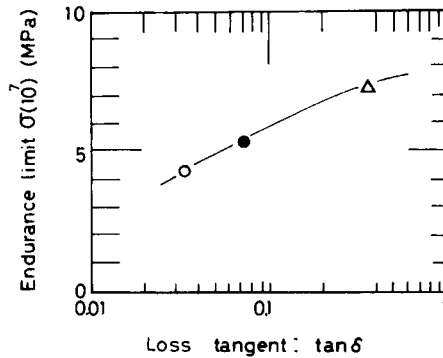


Fig. 7. Relationship between endurance limit and  $\tan \delta$ : (○) Epikote 828-Versamid 140; (●) Epikote 828-CTBN-Versamid 140; (Δ) Epikote 828-Versamid 115.<sup>3,4</sup>

becomes gradually steep with the increase in the stress cycles in contrast to the  $S-N$  relation for the nonimpact fatigue. In this work, such a trend in the fatigue strength behavior was also revealed for several kinds of adhesives.

Furthermore, it is observed that the stress cycles dependence of impact fatigue strength for the adhesive joint modified with CTBN is small compared with another adhesive joints. We may conclude that the CTBN modification not only raises the internal friction loss in the adhesive but also improves the impact fatigue strength.

The effect of CTBN modification would be attributed to the dispersion of rubber particles which absorb the impact energy to adhesive joint and inhibit the formation and growth of voids. This is valuable information about adhesive joints under the impact load conditions.

### Effect of Adhesive Thickness on the Fatigue Strength

It is well known that the adhesive strength decreases with decreasing the adhesive layer thickness in monotonic tensile test.<sup>15</sup> In this section, the effect of

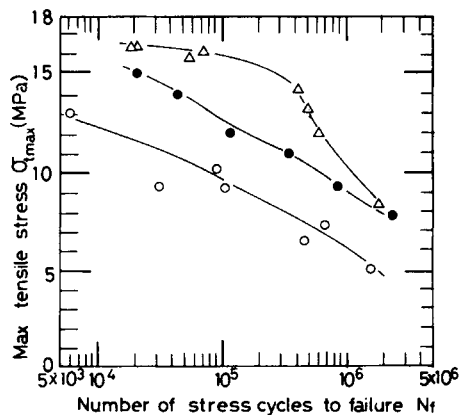


Fig. 8. Results of impact fatigue tests (adhesive thickness  $t = 0.1$  mm): (●) Epikote 828-CTBN-Versamid 140; (○) Epikote 828-Versamid 140; (Δ) Epikote 828-Versamid 115.<sup>3,4</sup>

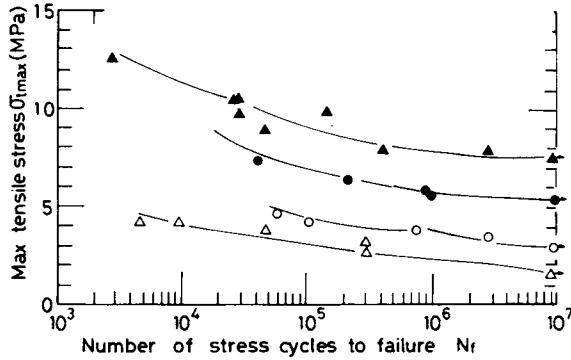


Fig. 9. Effect of adhesive thickness on fatigue strength under nonimpact stress conditions: (●,○) Epikote 828-CTBN-Versamid 140; (▲,△) Epikote 828-Versamid 115.<sup>3,4</sup> Adhesive thickness  $t$  (mm): (●,▲) 0.1; (○,△) 0.5.

adhesive thickness on the fatigue strength is discussed for two types of adhesives under the impact and nonimpact stress conditions.

Figure 9 shows the results for the nonimpact stress condition. The figure indicates that the fatigue strength increases with decreasing the adhesive layer thickness. Such a strength behavior agrees well with that of the static strength. These static strength behaviors are attributed to the shear stress distribution along the bonding interface.<sup>15-17</sup> Taking into account the effect of the layer thickness on the fatigue strength, one can estimate that the degree of lowering the fatigue strength of the CTBN-modified adhesive joint is smaller than that of unmodified one. This suggests that the CTBN modification results in a reduction of the residual stress induced from the solidification shrinkage of adhesives.

Figure 10 shows the effect of adhesive thickness on the impact fatigue strength. As can be seen from the figure, the increase of the adhesive thickness brings about the drastic decrease in the impact fatigue strength in a relatively low stress cycles

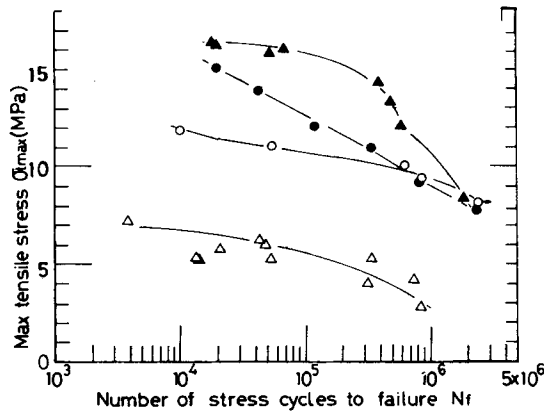


Fig. 10. Effect of adhesive thickness on fatigue strength under impact stress conditions: (●,○) Epikote 828-CTBN-Versamid 140; (▲,△) Epikote 828-Versamid 115.<sup>3,4</sup> Adhesive thickness  $t$  (mm): (●,▲) 0.1; (○,△) 0.5.

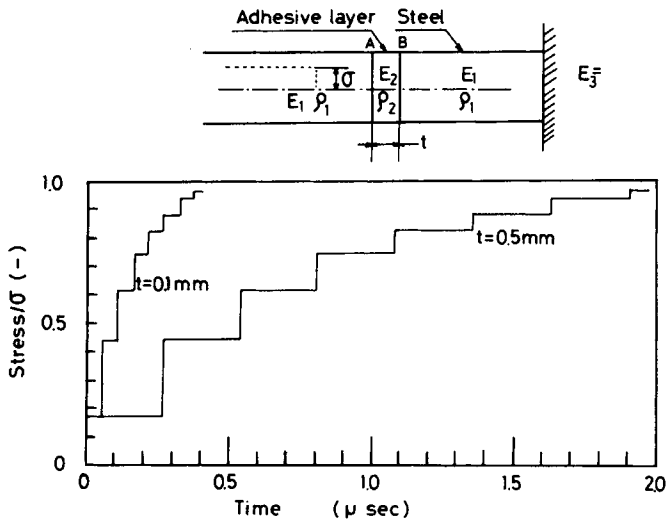


Fig. 11. Aspect of stress rising process.

range. Such a tendency also agrees with that for the nonimpact stress condition. But, the distinct stress cycles dependence of impact fatigue strength is observed for the thicker adhesive ( $t = 0.5$  mm), as are shown by the open symbols (O and  $\Delta$ ) in Figure 10. That is, the stress cycle dependence becomes gentle in accordance with increase in the adhesive thickness.

In the previous work,<sup>3</sup> we reported such a thickness dependence using the Epikote 828-Versamid 115 and explained such a phenomenon from the viewpoint of severity of the impact stress.<sup>3,4</sup> Also, such a fatigue behavior was revealed for different kinds of adhesive, as mentioned above. Here, to clarify the relation between severity of impact stress and adhesive thickness, a stress-time diagram at location A near the adhesive/adherend interface was obtained by a graphical calculation, as is shown in Figure 11. In this calculation, a step function for the stress, which has the magnitude of  $\sigma$ , was assumed as the input stress. Besides this, the material constants used are as follows: the Young's modulus  $E_1 = 2.06 \times 10^5$  MPa (steel) and  $E_2 = 1.47 \times 10^4$  MPa (adhesive), the density  $\rho_1 = 8.0$  g/cm<sup>3</sup> (steel) and  $\rho_2 = 1.06$  g/cm<sup>3</sup> (adhesive). Figure 11 indicates that stress rise velocity decreases with increasing the adhesive layer thickness. In addition, the behavior of the fatigue strength under impact stress conditions is likely to become near that under nonimpact stress, with increasing the adhesive thickness; the thickness dependency of impact fatigue strength can be reasonably explained.

Furthermore, as mentioned above, for the adhesive joint modified with CTBN, the stress cycles dependence in impact fatigue and the degree of fatigue strength lowering with increase in the adhesive thickness are smaller than unmodified ones. Thus, the following peculiar behavior seems to result. The impact fatigue strength for the 0.5 mm adhesive thickness joint is larger than for the 0.1 mm thickness joint at stress cycles more than  $8 \times 10^5$ . From such a discussion, a conclusion would be drawn that the relation between the fatigue strength and the adhesive layer thickness under the impact stress conditions is different from that for the nonimpact one.



## CONCLUSIONS

To improve the impact fatigue strength of the adhesive joint, an epoxy-polyamide adhesive was modified with CTBN. Impact fatigue characteristics were discussed in comparison with those under the nonimpact stress conditions.

Major conclusions obtained in this study are summarized as follows.

1. Under the nonimpact stress conditions, it is possible to correlate the fatigue strength with the internal friction loss.

2. In relatively low stress cycles range, the impact fatigue strength was higher than that in nonimpact fatigue. But, the slope of the  $S-N$  curve under the impact stress conditions became gradually steep with increase in the stress cycles contrary to the  $S-N$  relation in nonimpact fatigue.

3. The stress cycles dependence of impact fatigue strength for the adhesive joint modified with CTBN was small compared with nonmodified adhesive joints. That is, the impact fatigue strength is improved by CTBN modification.

4. The fatigue strength under the nonimpact stress conditions increased with decreasing the adhesive layer thickness, whereas the impact fatigue strength increased in relatively low stress cycles range. But, under the impact stress conditions, the stress cycle dependence became gentle in accordance with increase in the thickness. Particularly, for the CTBN-modified adhesive joint, the impact fatigue strength for the 0.5-mm adhesive thickness joint was larger than that for the 0.1-mm adhesive thickness joint in the stress cycles range exceeding  $8 \times 10^5$ . Therefore, the relation between the fatigue strength and the adhesive layer thickness under the impact stress conditions is different from that for the non-impact one.

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