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Vibration damping properties of gradient polyurethane/vinyl ester resin interpenetrating polymer network

C.L. Qin [a,*], D.Y. Zhao [a], X.D. Bai [a], X.G. Zhang [b], B. Zhang [b], Z. Jin [a], H.J. Niu [a]

[a] College of Chemistry and Chemical Engineering, Heilongjiang University, Harbin 150080, PR China
[b] Heilongjiang Institute of Petrochemistry, Harbin 150040, PR China

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Abstract

In this paper, the vibration damping properties were measured by cantilever method with steel beams as substrate, gradient PU/VER (BMA) IPN as coatings and when used, polysulfide rubber modified epoxy resin without fillers and with common inorganic fillers and whisker crystals as constrained layer. The effects of the thickness ratio of damping layer and steel beam and the sequence of gradient coating on loss factor (η) of extensional damping structure were studied. The effects of the thickness ratio and the time interval of coating (the time difference between coating a layer and another layer) between constrained layer and damping layer on damping properties of constrained damping structure were detected. Modulus of constrained layer was further increased by adding common fillers and inorganic whisker crystals in order to increase η of overall structure. The results show that damping properties of the extensional damping structure with the thickness ratio of 2:1 and the sequence of 70:30–60:40–50:50 (on the steel beam, the first layer is IPN with the component ratio of 70:30, the second layer is 60:40 IPN and the third layer is 50:50 IPN), are better compared with the others. When the thickness of constrained layer and damping layer is respectively 1 mm and the time interval of coating is 3 h, the η of optimized constrained damping structure with 10% aluminum borate ($Al_{18}B_4O_{33}$) whisker crystal in the constrained layer at 2nd mode are higher than 0.14 from −20 to 55 °C and its η at 2nd mode is 0.32 at −20 °C. The binding condition between constrained layer and damping layer was observed by SEM.

Keywords: Vibration damping; Gradient IPN; Polyurethane; Vinyl ester resin

1. Introduction

The conversion of vibration energy to heat is of special interest in damping unwanted noise because a polymer, at its glass transition conditions and in contact with a vibration surface, rapidly converts the mechanical energy to thermal energy, thereby reducing the emitted noise [1]. The theoretical aspects of resonant vibration attenuation by coatings have been described by Ungar [2], who described two main types of coating configurations: extensional and constrained. An extensional damping treatment is a single layer coating on an elastic substrate (e.g., steel) in which energy dissipation (and consequent damping) evolves primarily from the flexural and extensional motions of the damping layer. A constrained layer treatment consists of a two-layer system on the substrate with a viscoelastic layer under a stiff constraining layer. The addition of the constrained layer produces a shear action within the viscoelastic layer as the composite panel vibrates. The shear action in combination with flexure and extension greatly increases the amount of energy dissipated per cycle over extensional systems.

IPN has been the promising technique of preparing materials with broad T_g ranges and excellent damping performances [3,4]. Gradient IPN, a mixture of crosslinked polymers in which the concentration of one network changes across the section of a sample, may be regarded as a combination of an infinite number of layers of IPN [5], and it was shown that gradient IPN had a maximum of mechanical loss tangent (tan δ) spanning a broad temperature range [6,7] and more excellent damping properties than IPN.

In recent years, there has been increasing interest in polymers for vibration and damping applications. Most of the research has concentrated on the damping properties of neat polymers [8], polymer foams [9], and IPNs [10,11]. For PU/VER IPN, a great deal of research has been concentrated on their synthesis,

* Corresponding author. Tel.: +86 451 8660 8131; fax: +86 451 8660 8131.
E-mail address: chuanliqin@sohu.com (C.L. Qin).

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morphology and mechanical properties, but seldom on their damping properties [12,13] and on the evaluation of the vibration damping efficiency of the IPN-laminated steel beams [14]. In the previous work [6], a series of polyurethane/vinyl ester resin (PU/VER) gradient IPNs having broad, useful temperature ranges of damping were synthesized by casting the mixture of different component ratios in a mold at various times. In this paper, the vibration damping properties were studied with steel beams as substrate, gradient PU/VER(BMA) IPN as coatings and when used, polysulfide rubber modified epoxy resin without fillers and with common inorganic fillers and whisker crystals as constrained layer. The effects of the thickness ratio of damping layer and steel beam and the sequence of gradient coating on loss factors (η) of extensional damping structure were studied. The effects of the thickness ratio, the time interval of coating (the time difference between coating a layer and another layer) between constrained layer and damping layer and Modulus of constrained layer on damping properties of constrained damping structure were detected. Furthermore, the binding condition between constrained layer and damping layer was observed by SEM.

2. Experimental

2.1. Materials

Phosphating agent, surface conditioning agent and phosphating accelerator were chemical pure and supplied by Harbin Huaxing Corporate. Bisphenol A epoxy resin E-51 was supplied by Wuxi Synthetic Resin Plant. Three kinds of polysulfide rubber (JLY-121, JLY-124, JLY155) were supplied by Jinxi Research Institute of Chemical Industry. Aluminum borate ($Al_{18}B_4O_{33}$) whisker crystal, calcium carbonate ($CaCO_3$) borate whisker crystal, calcium sulfate ($CaSO_4$) borate whisker crystal were supplied by Qingha Institute of Salt Lakes. $CaCO_3$ powder, Mica and wollastonite powder were supplied by Beijing Guoli Superfine Powder Company. Active diluents, polyamide curing agents and γ-aminopropyltriethoxysilane (KH550) coupling agent were respectively supplied by Beijing Yili Chemical Company, Tianjin Jindong Chemical Plant and Nianjing Shuguang Chemical Plant. Polyamide curing accelerator was prepared in our laboratory. Steel plates with 1 mm thickness were purchased from Steel & Iron Factory. Other reagents were chemically pure and obtained from various suppliers. Materials of IPN damping layer were listed in the previous paper [6].

2.2. Preparation of extensional and constrained IPN beams

2.2.1. Preparation of extensional IPN beams

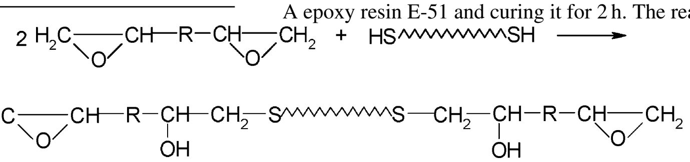
Before the preparation of IPN beams, steel beams (20.0 cm × 1.0 cm × 0.1 cm) were pretreated in order to improve the adhesive attraction between IPN materials and steel beams. First, the phosphating process was adopted as follows. 25 ml Phosphating agent, 1–1.2 g $NaCO_3$ and 1–1.5 ml phosphating accelerator were dissolved in 1000 ml water and the free acidity of the solution was remained at 0.5–0.8, total acidity was remained at 18–22. If the free acidity was lower than 0.5, phosphating solution was added and if the free acidity was higher than 0.8, $NaCO_3$ was added. Second, the steel beam was coated with KH550 and dried at low temperature.

The structure of extensional IPN beams is illustrated in Fig. 1. The preparation of the gradient IPN mixture was presented in the previous paper [6].

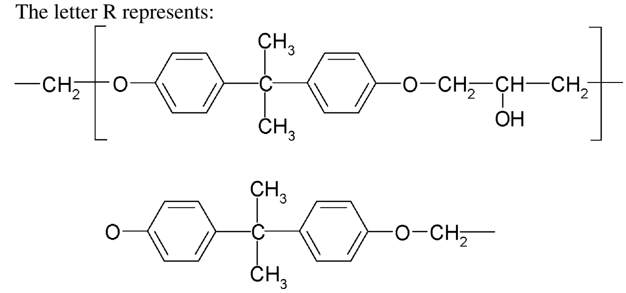
The steel beam was coated with gradient IPN mixture with different component ratios at the time interval of 3 h which indicates that another layer is coated when a layer has cured for 3 h. And the extensional beams were obtained by curing the samples at room temperature.

2.2.2. Preparation of polysulfide rubber modified epoxy resin materials as constrained layer

The prepolymer was prepared by mixing polysulfide rubber and Bisphenol A epoxy resin E-51 and curing it for 2 h. The reaction equation is as follows:



The letter R represents:



A component was the mixture of the prepolymer and active diluents and B component was the mixture of polyamide curing agents and curing accelerator. Both A and B component were preheated to 60 °C in order to lower their viscosity and then they were mixed and cured at room temperature to form the product. In order to improve its mechanical properties, common inorganic fillers and whisker crystals were added. First, the common inorganic fillers and whisker crystals were dried for several hours at certain temperature and then were stirred for an hour in the solution of KH550 and alcohol. Last the mixture was separated by the method of filtering and drying. In the other method, the inorganic fillers and whisker crystals were mixed with active diluents and polysulfide rubber modified epoxy resin to form A component and the other process was the same as that of polysulfide rubber modified epoxy resin.

2.2.3. Preparation of constrained IPN beams

First the gradient IPN coating was prepared on the surface of pretreated steel beam. Then the mixture of polysulfide rubber modified epoxy resin was added on the IPN coating after the last layer of gradient IPN polymerized for a period of time. Last the whole material cured to form the constrained IPN beam at room temperature and normal pressure. The structure is also shown in the Fig. 1.

2.3. Measurements

Mechanical properties were measured on a INSTRON 4467 tensile tester with extension rate of 100 mm/min. The samples were the dumbbell shape in accordance with GB1040-79.

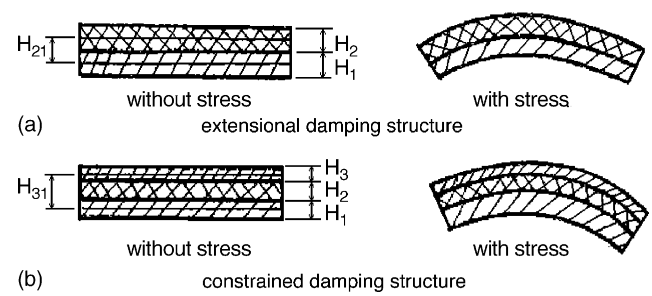


Fig. 1. The diagram of extensional and constrained damping structures. H_1: the thickness of substrate, H_2: the thickness of damping layer, H_3: the thickness of constrained layer, H_{21}: the distance between the center lines of damping layer and substrate, H_{31}: the distance between the center lines of constrained layer and substrate.

Table 1
Devise of orthogonal experiment and the experimental results

No.	The type of polysulfied rubber	The content of polysulfied rubber (%)	The mass ratio of polyamide curing agents and accelerator	Young's modulus (MPa)
I	1(121)	1(5)	1(15:15)	882.2
II	1(121)	2(10)	2(30:15)	790.5
III	1(121)	3(20)	3(45:15)	320.0
IV	2(124)	1(5)	2(30:15)	792.6
V	2(124)	2(10)	3(45:15)	349.0
VI	2(124)	3(20)	1(15:15)	706.5
VII	3(155)	1(5)	3(45:15)	391.5
VIII	3(155)	2(10)	1(15:15)	768.6
IX	3(155)	3(20)	2(30:15)	439.1
K1	1992.7	2066.2	2357.4	
K2	1848.2	1908.2	2022.2	Sum = 5440.1
K3	1599.2	1466.7	1060.6	
R	393.5	600.6	1296.8	
Optimized formulation	1(121)	1(5)	1(15:15)	882.2

SEM observations were carried out using a JEOL JXA-840 apparatus type. The samples was prepared by fracturing it in liquid nitrogen in the direction of thickness and applying a conducting gold coating in order to reduce the charging effect.

The vibration damping properties in terms of η were measured by cantilever method with a set of devices which include a Acceleration Transducer Type BK8307, a Charge Amplifier Type BK2635, a Dynamic Signal Analyzer Type HP3562A, a Power Amplifier Type BK2706 and a exciter transducer Type BK4810. The samples were steel beams coated with gradient IPN or gradient IPN and polysulfide rubber modified epoxy resin as mentioned above. The samples were placed in an environmental chamber operated over the temperature range −20 to +50 °C. The η was determined from the sharpness of the resonance curves $\eta = \Delta f/f_n$ in which f_n is the undamped resonance frequency of resonance mode n and Δf is the band width 3 dB down from the nth resonance peak.

3. Results and discussion

3.1. Mechanical properties of constrained layer materials

3.1.1. Mechanical properties of polysulfied rubber modified epoxy resin

The polysulfied rubber modified epoxy resins were prepared by orthogonal experiment as shown in Table 1 and the results are shown in Table 1 (in this paper, all the contents are mass content).

As shown in Table 1, in the range of selective factors and levels, the mass ratio of polyamide curing agent and polyamide curing accelerator is the most important, the amount of polysulfied rubber is the less important and the type of polysulfied rubber is the lest important. We can conclude that polysulfied rubber modified epoxy resin with greater modulus can be synthesized with polysulfied rubber with lower average molecular weight, smaller amount of polysulfied rubber and polyamide curing agents. The mechanical properties of the optimized material and pure epoxy resin are shown in Table 2. As shown in Table 2, both the modulus and toughness of the optimized polysulfied rubber modified epoxy resin is higher than pure epoxy resin and the mechanical properties present synergism.

3.1.2. Mechanical properties of polysulfied rubber modified epoxy resins with fillers

In order to further improve the modulus of constrained layers, common inorganic fillers were added to the optimized polysulfied rubber modified epoxy resin. The Young's modulus of polysulfied rubber modified epoxy resins with different kinds of inorganic fillers and different ratios is showed in Table 3.

As shown in Table 3, the type, shape, size and content of fillers have great influence on the properties of the composites. In the range of selective factors and levels, the modulus of the composites increases with the increase of the content for every kind of filler. In this paper, the maximum content is 50% because the viscosity of mixture is too high to handle when the content is higher than 50%.

Although its size is big, the effect of mica on modulus improvement of matrix is better than the other fillers because the sheet filler has high specific surface area. But the effect of sheet mica is not perfect because the modulus of matrix is just improved to certain content when the content is the maximum of 50%. The whisker crystal can evidently improve the modulus of resins according to the literature [15]. In this paper, cheap $CaCO_3$ borate whisker crystal, $CaSO_4$ borate whisker crystal and $Al_{18}B_4O_{33}$ whisker crystal with the high cost performance were chosen as the fillers to improve the modulus of matrix. The Young's modulus of polysulfied rubber modified epoxy resin

Table 2
Mechanical properties of polysulfied rubber modified epoxy resin and pure epoxy resin

Mechanical properties	Polysulfied rubber modified epoxy resin	Pure epoxy resin
Elongation at break (%)	16.5	11.1
Tensile strength (MPa)	75.2	78.0
Elongation at maximum load (%)	16.5	11.1
Tensile strength at maximum load (MPa)	75.2	78.0
Young's modulus (MPa)	882.2	820.0
Impact strength (KJ m^{-2})	25.1	14.4

Table 3
Young's modulus of polysulfied rubber modified epoxy resin with common fillers

Filler	Shape	Size (mesh)	Content (%)	Young's modulus (MPa)
$CaCO_3$ power	Granular	325	10	916.2
			20	961.1
			30	1020.0
			40	1122.0
			50	1241.0
Mica	Sheet	250	10	997.5
			20	1094.0
			30	1166.0
			40	1336.0
			50	1378.0
Wollastonite powder	Acicular	1250	10	910.3
			20	958.9
			30	998.6
			40	1080.5
			50	1154.0

with different types and ratios of whisker crystals is showed in Table 4.

As shown in Table 4, the $CaCO_3$ borate whisker crystal has better effect on the improvement of modulus than $CaCO_3$ powder, which affirms the predominance of whisker crystals on mechanical properties. In contrast, $Al_{18}B_4O_{33}$ whisker crystal is the best and chosen as the filler to improve the modulus of constrained layer because the modulus of matrix is improved by 47% when its content is only 10%.

3.2. Damping properties of extensional IPN damping structure

3.2.1. Damping properties of extensional IPN damping structure with different thickness ratios

The curves of η of extensional IPN damping structure and thickness of IPN damping layer are shown in Fig. 2 (the IPN with the component ratios of 60:40 is the damping layer).

As shown in Fig. 2, η of the composite structure decreases with the increase of resonance mode (resonance frequency) and its damping properties are good at 2nd mode (low frequency). According to the curves of η and structure parameters [16], η continuously increases to a boundary value with the increase of the thickness. But in the range of selected thickness, η linearly increases with the increase of the thickness, which is due to the fact that the ratios of modulus of the prepared IPN materials and modulus of the selected steel beams are low and the selected ratios of their thickness are in the linear range of the curves of η and structure parameters (the operation of increasing the ratio of thickness continuously is difficult to realize in the practical condition).

According to GB/T 18258-2000, the recommended ratio of the thickness between damping layer and steel beam is 2:1. The η of the composite structure is high when the ratio of thickness is 2:1 and the curves in Fig. 2 bend downwards and the cost performance reduces when the ratio of thickness is 4:1 and 6:1. So the optimized ratio of thickness in this paper is 2:1.

3.2.2. Damping properties of extensional IPN damping structure with different sequence of gradient coating

The damping data of extensional IPN damping structure with different sequence of gradient coating are shown in Table 5. As

Table 4
Young's modulus of polysulfied rubber modified epoxy resin with whisker crystals

Whisker crystal	Content (%)	Young's modulus (MPa)
$Al_{18}B_4O_{33}$	10	1298.0
	20	1537.0
	30	2041.0
$CaSO_4$	10	892.1
	20	1182.0
	30	1283.0
$CaCO_3$	10	931.1
	20	1037.0
	30	1206.0

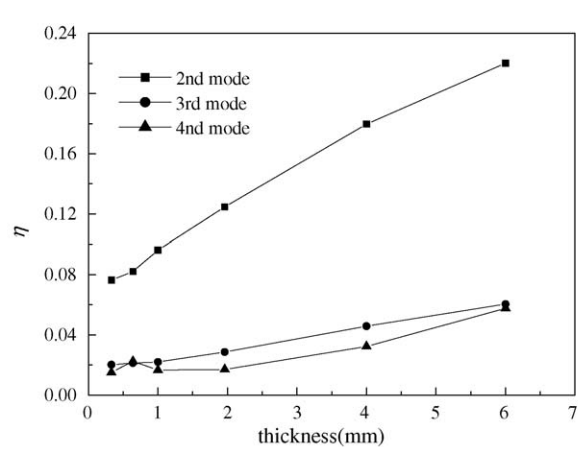


Fig. 2. The effect of thickness of damping layer on η at 2nd, 3rd and 4th mode.

Table 5
Damping properties of extensional damping structure with different coating sequence

Sequence of coating	Mode	f_n (Hz)	f_1 (Hz)	f_2 (Hz)	η
50:50–60:40–70:30	2nd	146.3	135.606	156.548	0.1431
50:50–60:40–70:30	3rd	390.7	386.387	395.352	0.0229
50:50–60:40–70:30	4th	750.7	746.768	757.53	0.0143
70:30–60:40–50:50	2nd	141.4	130.248	151.55	0.1507
70:30–60:40–50:50	3rd	381.6	377.498	386.206	0.0229
70:30–60:40–50:50	4th	731.4	726.478	737.429	0.0150

Note: $\Delta f = f_2 - f_1$.

shown in Table 5, the gradient IPN damping structure with the component ratio of 70:30–60:40–50:50 (on the steel beam, the first layer is IPN with the component ratio of 70:30, the second layer is 60:40 IPN and the outer layer is 50:50 IPN), has high η at every mode. It is due to the fact that the modulus of whole gradient IPN materials is same whatever sequence of coating they are and when the steel beam vibrates, the relative deformation of the gradient IPN damping layers with the outer layer of 50:50 IPN occur which generates additional energy dissipation and improves the damping effect.

3.3. Damping properties of constrained IPN damping structure

3.3.1. Damping properties of constrained IPN damping structure with different thickness ratios

In the range of 1000 Hz, the curves of amplitude frequency have four vibration modes measurable in this study. The η of 2nd mode, 3rd mode and 4th mode is studied because 1st mode is not obvious. The curves of η of composite structure and thickness of constrained layer are shown in Figs. 3–5 (the gradient IPN with the component ratios of 70:30–60:40–50:50 and the thickness of 1 and 2 mm is respectively the damping layer).

As shown in Figs. 3–5, η decreases with the increase of the mode or frequency and the composite structure has good damping properties at 2nd mode or low frequency. On condition that the thickness of damping layer is invariable, the η of composite structure increases with the increase of the thickness of constrained layer. As a whole, the η of composite structure increases most when the thickness of constrained layer is 1 mm and it increases less when the thickness of constrained layer is higher than 1 mm no matter what thickness of damping layer is. So the thickness of constrained layer is fixed at 1 mm considering cost performance.

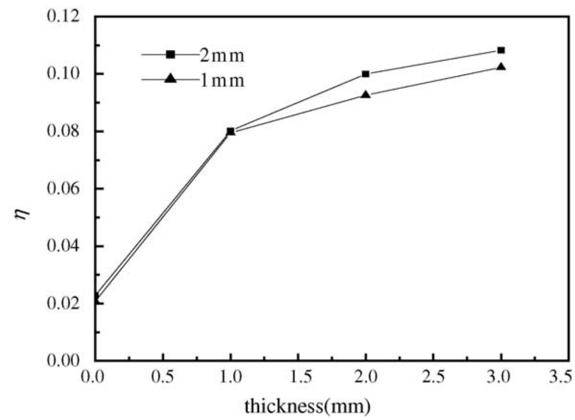


Fig. 4. The effect of thickness of constrained layer on η at 3rd mode.

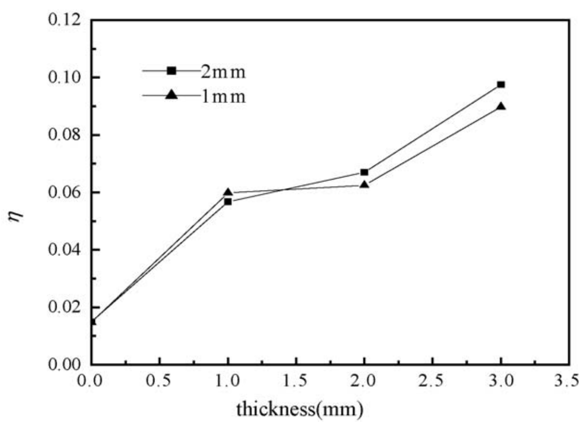


Fig. 5. The effect of thickness of constrained layer on η at 4th mode.

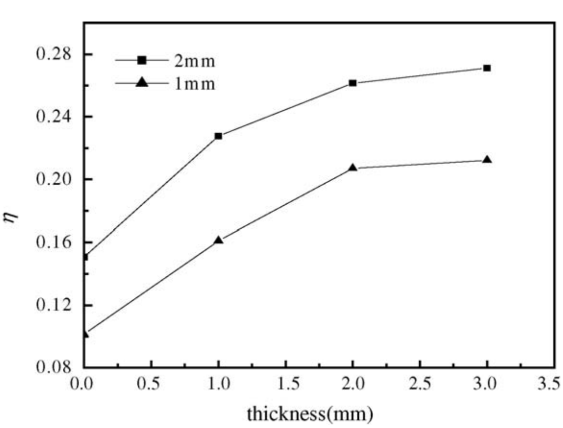


Fig. 3. The effect of thickness of constrained layer on η at 2nd mode.

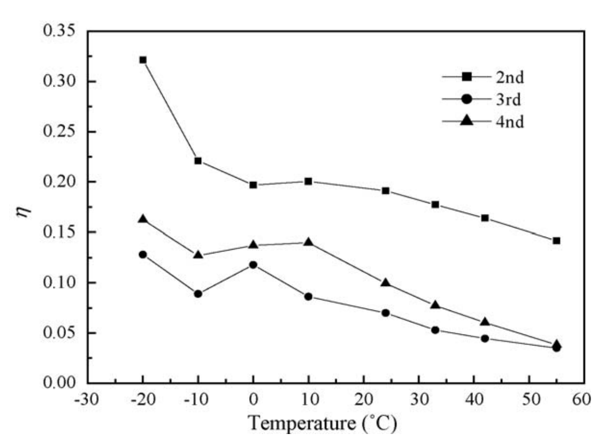


Fig. 6. Temperature dependence of η at the 2nd mode.

Table 6
η of composite structure with different time intervals of coating

Interval of coating(h)	Mode	f_1 (Hz)	f_n (Hz)	f_2 (Hz)	η
1.5	2nd	168.08	194.90	221.09	0.2720
	3*rd*	413.02	450.00	483.89	0.1575
	4*th*	741.80	796.10	828.68	0.1091
3	2nd	156.55	184.90	216.19	0.3280
	3*rd*	398.15	432.10	463.25	0.1507
	4*th*	726.20	773.50	804.59	0.1026
24	2nd	142.80	164.90	186.9	0.2614
	3*rd*	391.94	412.80	433.22	0.1000
	4*th*	732.79	760.70	783.74	0.0670

3.3.2. Damping properties of constrained IPN damping structure with different time interval of coating

The η of composite structure with different time intervals of coating is shown in Table 6 (the gradient IPN with the component ratios of 70:30–60:40–50:50 and both the thickness of the damping layers and constrained layer are 2 mm).

As shown in Table 6, when the time interval of coating is 3 h, the mixture of constrained layer is added on the damping layer which is just at gelation, the damping properties of the damping structure are excellent. When the time interval of coating is short (1.5 h) or long (24 h), the binding condition between constrained and damping layers is affected and further the dissipative energy by shear deformation is also affected. The conclusion is verified by the microstructure in the following.

3.3.3. Damping properties of constrained IPN damping structure with constrained layers of different modulus

The η of the polysulfide rubber modified epoxy resin containing 10% $Al_{18}B_4O_{33}$ whisker crystal with high modulus is compared with that containing no filler with low modulus (the time interval of coating is 3 h). As shown in Table 7, the constrained damping structure with pure polysulfide rubber modified epoxy resin as constrained layer, whose thickness of constrained layer and extensional layer is respectively 1 mm, has higher η than extensional damping structure with the damping layer of 2 mm thickness which has the same composition as the former. We can conclude that the constrained layer greatly contributes to better damping properties of the whole damping structure. As we expected, the damping properties are obviously improved with the increase of constrained layer modulus, particularly the damping properties in the lower frequency has higher improvement.

Table 7
η of composite structures with constrained layers of different modulus

Filler	Mode	f_1 (Hz)	f_n (Hz)	f_2 (Hz)	η
Without fillers	2nd	154.15	168.5	181.31	0.1611
	3*rd*	419.6	437.1	454.36	0.0795
	4*th*	788.38	814.2	837.12	0.0599
10% $Al_{18}B_4O_{33}$ whisker crystal	2nd	160.04	177.1	193.9	0.1912
	3*rd*	418.37	440.6	462.23	0.0995
	4*th*	784.62	814.2	839.05	0.0699

3.3.4. Damping properties of constrained IPN damping structure at different temperatures

In order to exactly know the damping properties of polymer materials, their tan δ should be studied in the temperature range of practical application. By the optimization of all experimental conditions at room temperature, the optimized constrained IPN damping structure with the time interval of 3 h, which has the constrained layer of polysulfide rubber modified epoxy resin with 10% $Al_{18}B_4O_{33}$ whisker crystal and the thickness of 1 mm and the damping layer with the thickness of 1 mm, is selected to study the damping properties at different temperatures by the method of cantilever. The results are shown in Fig. 6.

As shown, the shape of η–T curves at every mode is the same as that of the gradient IPN materials with the same damping layer in the previous paper (Fig. 7). Its η at 2nd mode is higher than 0.14 in the temperature range between −20 and 55 °C and its damping properties are excellent in the low frequency.

3.4. Binding condition between constrained layer and damping layer and relation of the binding condition and damping properties

As we known, the reason that the constrained layer greatly contributes to better damping properties of the whole damping structure is that the constrained layer produces additional shear action within the viscoelastic layer as the composite panel vibrates. Besides the modulus of constrained layer, the time interval of coating between constrained layer and damping layer can have influence on binding condition between two layers which further influences the shear action of constrained layer on damping layer them and the damping property improvement of the whole damping structure.

In order to observe the binding condition between constrained layer and damping layer, SEM is adopted to study the fracture surface of the composite materials composed of constrained layer and gradient IPN damping layer (the composite materials were prepared at room temperature and normal pressure and at different time intervals). SEM images are shown in Fig. 7.

As shown in Fig. 7a and b, when the time interval of coating between two layers is 1.5 h, broad interface appears between constrained layer and damping layer, and broad transitional region with different shade of color appears from interface to damping layer, which is due to short time interval between two layers. When the damping layer is just immobile, the mixture of constrained layer is added, a great deal of constrained layer mixture penetrates into the damping layer and cures, and the broad transitional region plays a role as constrained layer of lower modulus which results in weak shear action between two layers and poor damping properties of the whole structure. When the time intervals of coating between two layers are 3 and 24 h, no obvious transitional regions appear. But as shown in Fig. 7d in the condition of higher magnification, a narrow transitional region appears in the sample with the time interval of 3 h. In the transitional region, the binding strength is strong due to blend and interpenetration action of the constrained layer and damping layer, so the shear force between the two layers is big and the damping properties of the whole structure are excellent. When the time interval of coating is 24 h, no transitional region appears, the interpenetration action of the constrained layer and damping layer is weak, and the binding strength and shear force between layers are weaker than those of Fig. 7c and d, so the damping properties are poor. The results of SEM verify the data of Table 6 and the results also show that the damping properties of whole structure can be improved by adjusting the technology of coating to control the binding condition between constrained layer and damping layer.

4. Conclusions

The extensional and constrained IPN damping structures composed of optimized gradient IPN material as damping layer and when used, polysulfide rubber modified epoxy resin without fillers and with common inorganic fillers and whisker crystals as constrained layer were studied.

The optimized technique conditions are that high modulus polysulfied rubber modified epoxy resin with 10% $Al_{18}B_4O_{33}$ whisker crystal is used as constrained layer, the thickness of constrained and damping layer is respectively 1 mm, the time interval of coating between two lay-

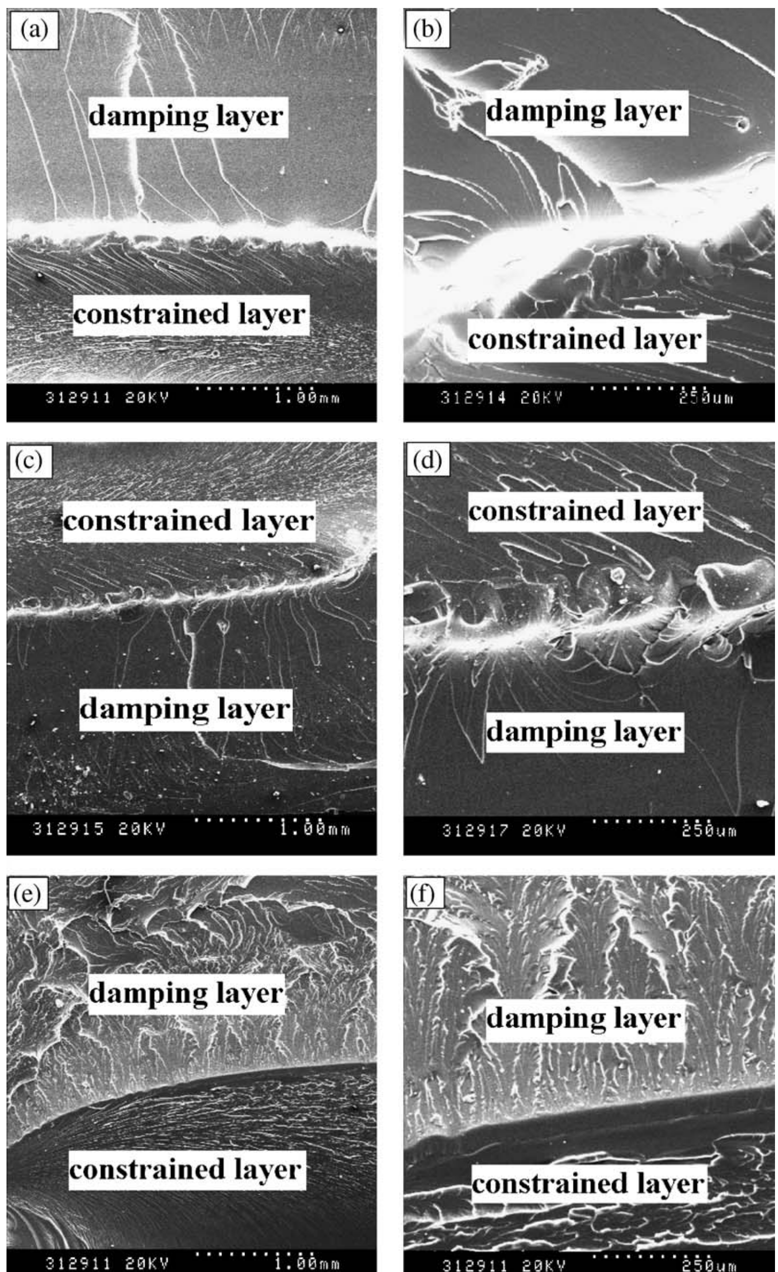


Fig. 7. SEM of composite materials with different time intervals of coating: (a) 1.5 h and lower magnification, (b) 1.5 h and higher magnification, (c) 3 h and lower magnification, (d) 3 h and higher magnification, (e) 24 h and lower magnification, (f) 24 h and higher magnification.

ers is 3 h. The constrained damping structure has greater η than free damping structure and it can dissipate more energy.

The time interval between constrained layer and damping layer has great influence on the interfacial binding conditions between two layers, which further affects the damping properties of the whole structure.

The detected results of optimized constrained IPN damping structure at different temperatures show that η–T curves of the composite structure are similar to tan δ–T curves of gradient IPN damping layer. Its η at 2nd mode is higher than 0.14 from −20 to 55 °C and its η at 2nd mode is 0.32 at −20 °C. The damping structure has excellent damping properties, especially at the low temperature and low frequency.

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