TEMPERATURE DEPENDENCY OF BOND STRENGTH OF POLYVINYL ACETATE EMULSION ADHESIVES FOR WOOD

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ABSTRACT

Bond strengths of PVAc emulsion adhesives were evaluated in the range from −130 to 140 C in both tensile shear and tensile cross-lap joints of wood. Both shear and tension bond strengths strongly depended on rheological properties and tensile strengths of adhesives over the entire temperature range, and the maximum peaks of each bond strength and the film strength were observed around the Tg of PVAc. Higher shear bond strengths were observed in the lower temperature range, while the other bond strengths declined markedly. This phenomenon was explained by the possible occurrence of mechanical interlocking at wood surface and difference of its effectiveness with the direction of stress on the joint. Furthermore, a high amount of grafting effectively fortified both the bond and film strengths of adhesives throughout the temperature range investigated.

Keywords: Temperature dependency, adhesives, bond strength.

We have previously investigated the influences of both chemical and physical elements of poly(vinyl acetate) (PVAc) emulsion adhesives, prepared by a combination of poly(vinyl alcohol) (PVA) emulsifier and the redox system initiator, on the bond strength of wood cross-lap joints (Motohashi and Tomita 1979, 1980; Motohashi et al. 1982). Among all the elements specified in our works, the grafting of vinyl acetate monomer on PVA, used as a surfactant at the time of synthesis, was the most significant one. The higher amount of the grafting was concluded to promote strong cohesion between the PVAc- and PVA-phases after removal of water from glueline and to enhance the ultimate strength of adhesive film.

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Bond strength was also strongly dependent on tensile strength of films in connection with the amount of grafting.

However, these evaluations were performed only at a constant temperature of 23 C. PVAc emulsions are thermoplastic polymers, as it is well known, which display glass transition temperature (Tg) near room temperature. Their mechanical properties change to a great extent by a slight difference of temperature in the region of the Tg. Therefore the practical bond quality must be strongly dependent on temperature.

Many reports can be found of research on the relations between bond strength, test-temperature, and test-speed (Kaelble 1969; Gent and Petrich 1969; Fukuzawa 1969). Adherends used in these reports, however, were not wood and bond strength was generally evaluated only by a peel test. On the other hand, there are few reports concerning the temperature dependency of wood bending. Chow examined the relation between durability and softening temperature of adhesives (Chow 1973), and Bergin and Chow reported that strength retention of PVAc emulsion adhesives at elevated temperature depended greatly on the ash content of the adhesives (Bergin and Chow 1974). Further, Steiner and Chow evaluated the low-temperature durability of many common wood adhesives including a cross-linked PVAc emulsion in a cyclic-exposure test (Steiner and Chow 1975).

Mizumachi and others have separately reported that the strength of bonded wood joints is dominated by the rheological properties of adhesives such as the modulus and the dynamic mechanical property (Mizumachi et al. 1979, 1980). In these works a series of adhesives gradually differing in mechanical properties were prepared by copolymerization or blending of more than two polymers with different glass transition temperatures. Their bond strengths were measured at a constant temperature. In the case of completely miscible blends of poly(vinyl chloride) and acrylonitrile-butadiene copolymer, the temperature corresponding to the absorption peak in loss modulus at 110 Hz, $T(E''_{max})$, could be made to vary at any levels between −9 and 70 C, depending on the blend ratio.

On the other hand, the continuous change of rheological property of polymers can be easily attained by temperature change without any chemical reactions. As merits of this method, all test specimens can be prepared under the same conditions, and all variable chemical factors concerning bonding conditions and nature of adhesives can be excluded.

From this background, we have undertaken to evaluate bond strength over a broad temperature range to obtain information about temperature effects on the mechanical properties of PVAc emulsion adhesives. Simultaneously the effects of an amount of the grafting on mechanical properties were also investigated using two emulsions with differences in grafting efficiency: one of 54.4% efficiency and the other of 12.2%. The bond strength was examined by both tensile shear and cross-lap tension, the latter of which has been reported as a sensitive method for testing common wood adhesives (Marra 1955a, b, c).

We believe that these works will promote the practical application of adhesives in the ordinary temperature range from −30 to 50 C, which arise by changing meteorological conditions and geographic location. Furthermore, we believe the data obtained outside this practical temperature range will shed light on the mechanism of wood bonding and also assist the development of chemical and physical modifications of wood adhesives.

TABLE 1. *Chemical properties of PVAc emulsion adhesives.*

Emulsion	Monomer conversion (%)	Grafting efficiency[1] (%)	Molecular weight of benzene soluble part[2]		
			$\bar{M}n \times 10^{-4}$	$\bar{M}w \times 10^{-4}$	$\bar{M}w/\bar{M}n$
A	98.7	54.4	5.12	20.8	4.06
B	96.2	12.2	2.18	6.52	2.98

[1] Percentage of unextracted PVAc amount with benzene to total PVAc in dried film.

[2] Calculated from gel permeation chromatogram. $\bar{M}n$: number-average molecular weight, $\bar{M}w$: weight-average molecular weight.

EXPERIMENTAL

PVAc emulsion adhesives

Partially saponified PVA (degree of polymerization 1,500, degree of saponification 86.6–89%) was used as an emulsifier. The redox system of tartaric acid-hydroperoxide was employed to initiate polymerization. The solid content of each emulsion was about 50%, and the weight ratio of PVAc to PVA was about 9:1. Two emulsions largely differing in their grafting efficiency were designated as Emulsion A and Emulsion B. The details of emulsion synthesis and characterization were described in a preceding paper (Motohashi et al. 1982). Some chemical properties of Emulsion A and B are shown in Table 1.

Measurement of dynamic mechanical properties of adhesives

The specimens were prepared by casting wet films of emulsion on Teflon sheets, drying at room temperature, and then heating at 110 C for 24 h after cutting specimens 3 × 10 × 50 mm. The dynamic mechanical properties were measured with a Rheovibron DDV-III (Toyo Baldwin Co. Ltd.) at 3.5 Hz in the range from −150 to 90 C. The rate of temperature increase during the tests was 1 C/min. The properties measured with the Rheovibron were storage modulus (E′) and loss modulus (E″). The storage modulus is a measure of the stiffness of the materials, while the loss modulus is a measure of the amount of energy lost as heat during dynamic testing (Nielsen 1975).

Torsional braid analysis was carried out with a torsional pendulum apparatus (RESCA Co. Ltd.). About 100 mg of emulsion was brushed onto a glass braid. It was dried at atmospheric pressure and room temperature for two days, and then at 10^{-4}–10^{-5} mm Hg for two days. Results were plotted in terms of relative rigidity (G′) and G′ · λ, $G' = (Po/Pt)^2$ being used, where λ is logarithmic decrement and Po and Pt were frequency intervals at −150 and t C, respectively. The relative rigidity (G′) is proportional to the storage modulus (E′) and G′ · λ is also proportional to the loss modulus (E″).

Tensile test for adhesive films

Films were prepared as described above. After drying, dumbbell-shaped specimens were cut as in Fig. 1. The specimens were conditioned at 20 C–65% relative humidity for 30 days before test. Gage lines spaced 10 mm were marked on the necked down area of each specimen at the position shown in Fig. 1. A Tensilon tensile tester (Toyo Baldwin Co. Ltd.) equipped with a temperature control chamber was utilized for tension tests. Temperature control was performed by a combination of liquid nitrogen cooling and an electric heater. Target temperatures inside specimens could be attained within 30 min after setting. The initial distance

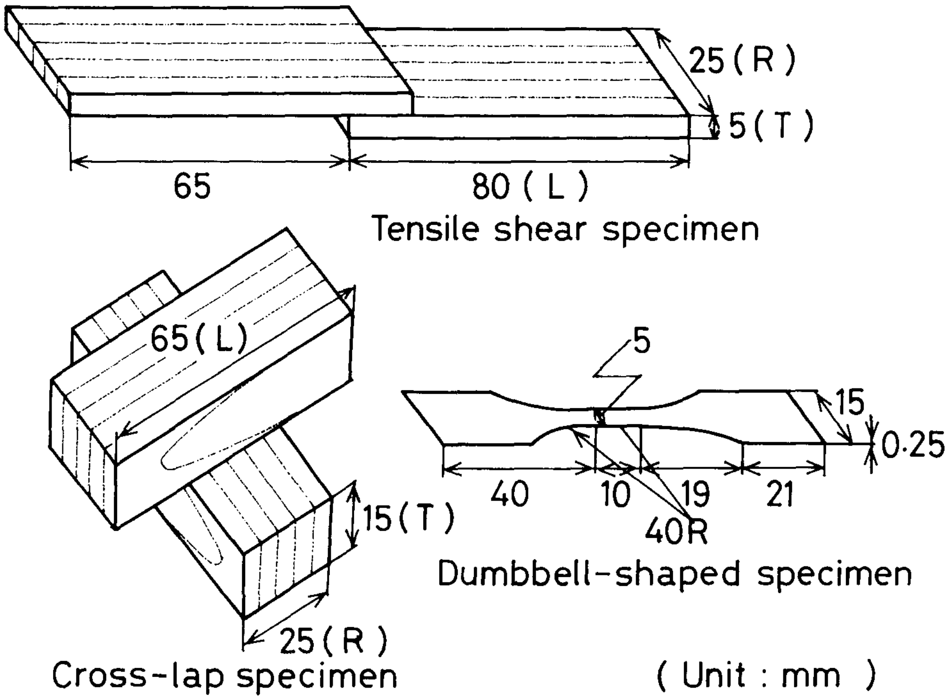

FIG. 1. Dimension of specimens.

between the jaws was 30 mm and the cross-head speed was 10 mm/min. The ultimate tensile strength was calculated on the basis of the original cross-sectional area of specimens tested in the low temperature range from −120 to 20 C, where elongation of specimens was negligible. In the temperature range from 30 to 120 C, where elongation was observed, the reduced cross-sectional area calculated from assuming that Poisson's ratio is 0.5 was used to calculate the ultimate tensile strength. Each strength in Fig. 4 is a mean of 15–20 specimens.

Tests of bond strength

Birch (*Betula Maximowiczii* Regel, moisture content 12%) was used for the adherends. Dimensions of the tensile shear and cross-lap specimens are given in Fig. 1. Open and closed assembly time during bonding were 0 and 2–5 min, respectively, and bonding pressure was 0.98 MPa. After bonding under pressure for 24 h, specimens were removed from the press and conditioned at 20 C and RH 65 for 30 days before test. The same tensile tester and cross-head speed were used as in the tensile film tests. Twelve specimens were tested at each temperature.

RESULTS AND DISCUSSION

Temperature dependencies of mechanical properties for PVAc emulsion adhesives

The dynamic mechanical property of polymer is generally dependent on the excitation frequency, but it is well known that the temperature of the loss modulus (E″) peak measured in the neighborhood of 1 Hz is nearly equivalent to glass

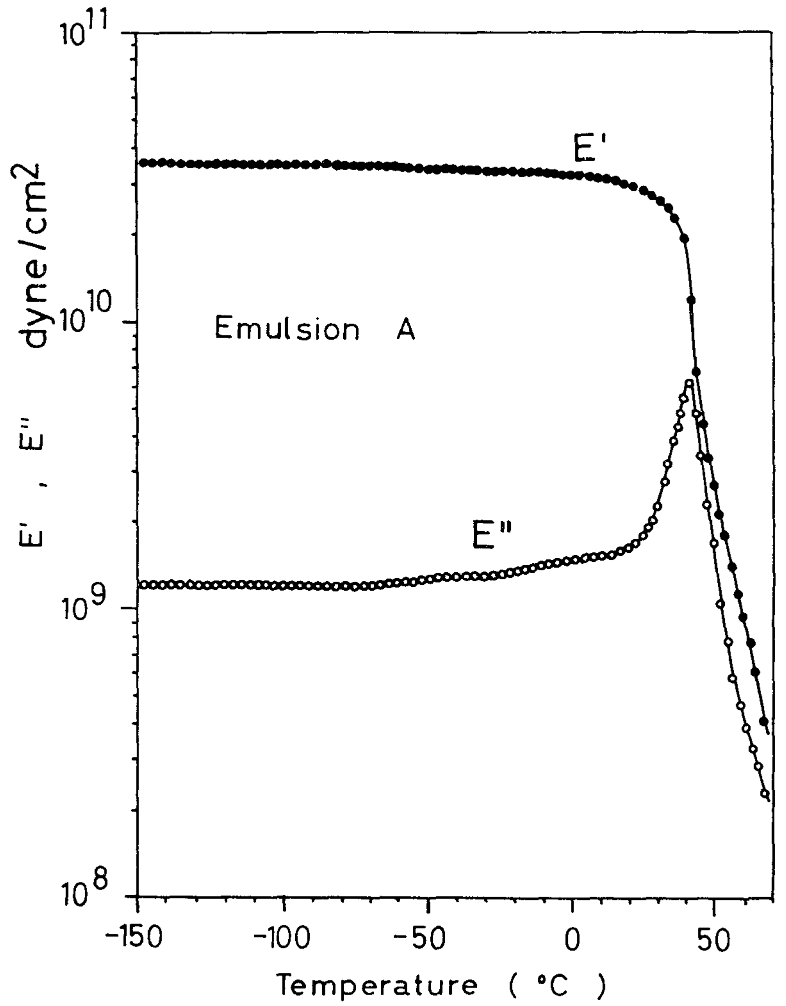

FIG. 2. Temperature dependence of storage modulus E' and loss modulus E'' of Emulsion A.

transition temperature determined from dilatometric method (Nielsen 1975). Figure 2 shows the temperature dependency of the dynamic mechanical property for Emulsion A at 3.5 Hz. The storage modulus (E') was constant between −150 and 30 C but dropped abruptly above 30 C because of the glass transition of PVAc. Correspondingly, the loss modulus was constant below 25 C and began to rise steeply above that temperature until a peak was observed at 41.5 C. Emulsion B was also investigated in this manner, but no distinct difference between the two emulsions was observed.

Although the emulsion films always contained 10% PVA as an emulsifier, the PVA glass transition was not detected by the Rheovibron as indicated by the absence of a secondary peak in Fig. 2. This is due to the large drop of storage modulus and elongation of specimen, which mask the smaller PVA effect. Two peaks in the function of $G' \cdot \lambda$, which may be considered proportional to loss

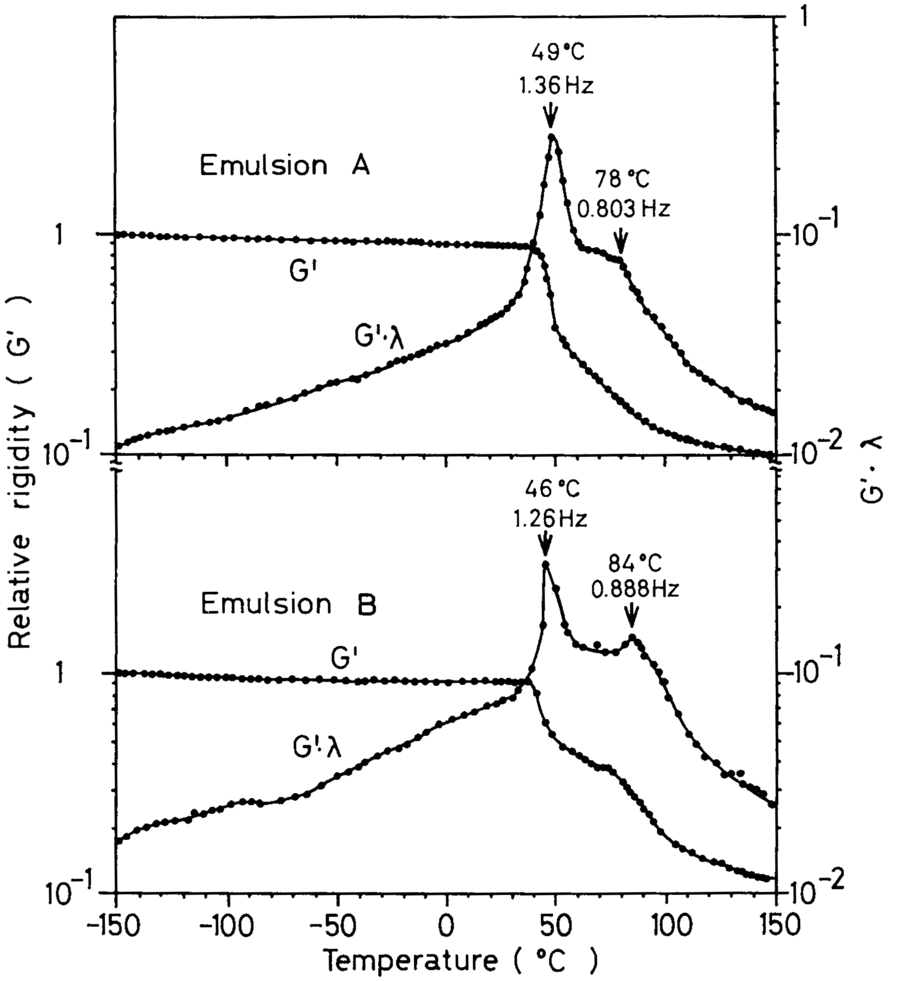

FIG. 3. Torsional braid analysis of two emulsion adhesives.

modulus (E″), were observed by the torsional braid analysis method as shown in Fig. 3. They are attributed to glass transitions of PVAc and PVA, respectively, from the lower temperature side. The appearance of two dispersion peaks in this dynamic measurement agrees with the electron microscope work of Bradford and Vanderhoff (1972), who originally pointed out the phase separation in PVAc emulsion films after removal of water. The PVA that coats the surface of emulsified PVAc particle forms a continuous phase and the PVAc a dispersed phase.

The relative rigidity (G′) of Emulsion B fell stepwise with elevation of temperature, showing a plateau near 75 C. On the other hand, that of Emulsion A fell continuously, no evident plateau. The dispersion peak due to PVA in G′·λ of Emulsion A shifted to lower temperature region by 6 C, and its intensity diminished, compared with Emulsion B. Furthermore, the temperature difference between two dispersion peaks of Emulsion B was larger by 9 C than that of Emulsion A—that is to say, two peaks tend to draw near each other in Emulsion

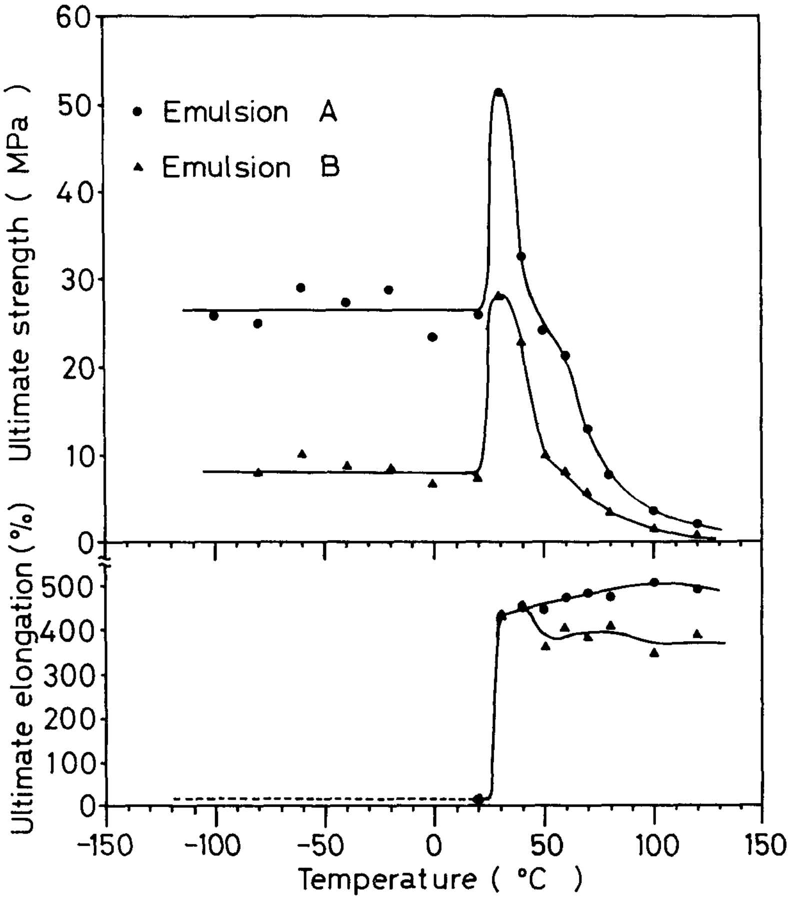

FIG. 4. Temperature dependence of ultimate tensile strength and ultimate elongation of two emulsion adhesive.

A. These phenomena strongly supported the supposition that a large amount of grafting between the two phases minimizes phase separation (Angelo et al. 1965; Uchida et al. 1972).

Figure 4 shows the temperature dependencies of ultimate tensile strength and ultimate elongation of adhesive films. Ultimate tensile strengths of both films were constant in the glassy state below or near 20 C, where little elongation was observed. In this region specimens are considered to break without undergoing a large deformation, as the micro-Brownian motions of backbone chains in the polymer are too slow to follow the rate of deformation. The film strength begins to rise steeply with increasing temperature from near 20 C and shows the maximum at 30 C. In this glass transitional region, the molecular motions are fast enough to follow the rate of deformation and so the deformation energy is transformed into molecular motions. Consequently the film strength and the elongation increase abruptly. In the rubbery state beyond the glass transition temperature, elongation was constant and the film strength dropped slowly with increasing

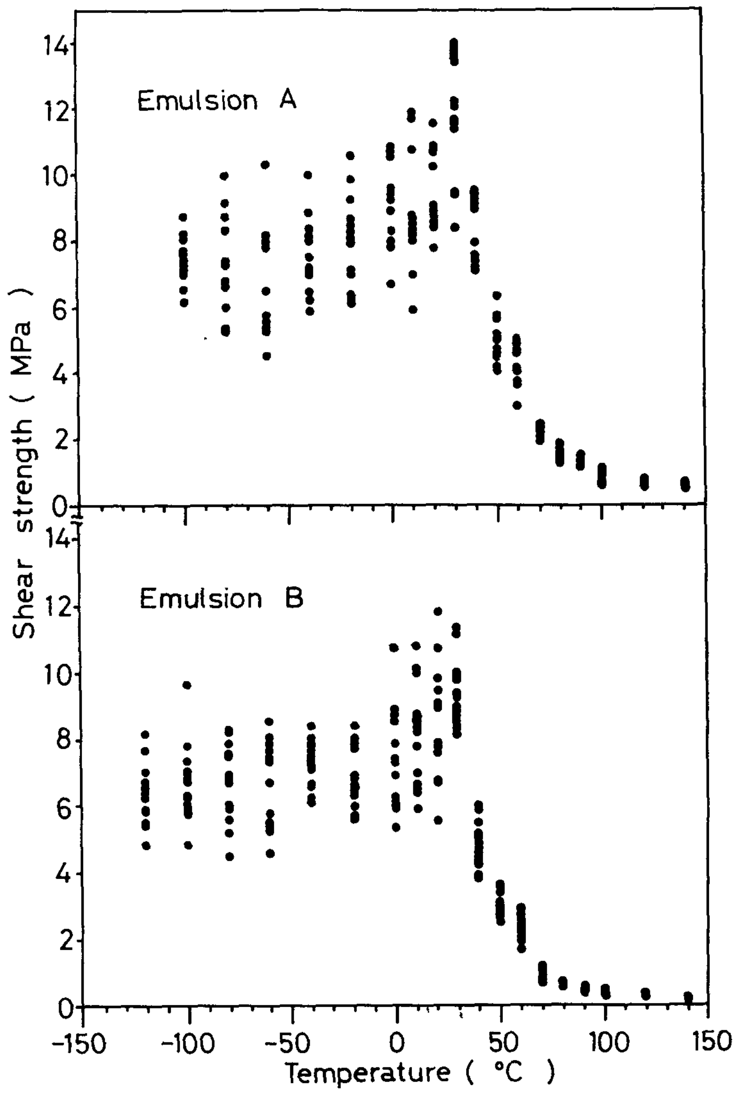

FIG. 5. Temperature dependence of tensile shear strength for two emulsion adhesives.

temperature. These results are well recognized as characteristic of amorphous polymers (Nielsen 1975).

In comparing the two emulsions, it must be noted that Emulsion A displayed higher ultimate strength than Emulsion B over the entire temperature range. This may well be explained by the difference in the amount of grafting between PVAc- and PVA-phase, and strongly supports the conclusion on phase structure derived from temperature dependency of dynamic mechanical property.

Temperature dependency of bond strengths

Results of the tensile shear tests over the entire temperature range are given in Fig. 5 and Table 2. Bond strengths of both adhesives were almost constant in

TABLE 2. *Average bond strength and average wood failure values for tensile shear specimens from −130 to 140 C.*

	Emulsion A				Emulsion B			
Temp.	$\bar{X}$[1] (MPa)	S[2] (MPa)	C.V.[3]	W.F.[4] (%)	$\bar{X}$ (MPa)	S (MPa)	C.V.	W.F. (%)
−120	—	—	—	—	6.43	0.925	0.14	35.0
−100	7.47	0.696	0.093	91.6	6.75	1.23	0.18	37.5
−80	7.27	1.49	0.20	37.9	6.56	1.11	0.17	20.2
−60	6.86	1.66	0.24	80.0	6.70	1.30	0.19	24.2
−40	7.50	1.18	0.16	77.5	7.29	0.727	0.10	16.7
−20	8.06	1.33	0.17	90.0	6.89	0.947	0.14	8.3
0	8.93	1.29	0.14	49.6	7.64	1.57	0.21	2.5
10	9.85	1.08	0.20	50.4	8.27	1.57	0.19	9.2
20	9.32	1.15	0.12	50.8	8.64	1.75	0.20	4.2
30	11.8	1.92	0.16	35.8	9.11	0.913	0.10	0
40	8.05	0.883	0.11	17.9	4.99	0.642	0.13	0
50	4.96	0.686	0.14	0	3.05	0.342	0.11	0
60	4.24	0.637	0.15	0	2.39	0.380	0.16	0
70	2.11	0.235	0.11	0	0.981	0.189	0.19	0
80	1.47	0.212	0.14	0	0.732	0.125	0.17	0
90	1.21	0.192	0.16	0	0.532	0.0625	0.12	0
100	0.874	0.189	0.22	0	0.445	0.111	0.25	0
120	0.713	0.114	0.16	0	0.353	0.0481	0.14	0
140	0.609	0.0645	0.11	0	0.259	0.0174	0.066	0

[1] Mean value calculated from twelve specimens.
[2] Standard deviation.
[3] Coefficient of variation.
[4] Wood failure.

the glassy state in the lower temperature region where molecular motions of adhesive polymers are frozen. As test-temperature was elevated toward the Tg of PVAc, bond strength gradually rose to a maximum value around 30 C, where micro-Brownian motions of backbone chain in PVAc begin abruptly. Then it slowly decreased as the temperature increased above 30 C. Cohesive fracture was predominant above 30 C. Although a significant difference of bond strength to temperature could not be observed between two adhesives over the entire temperature range, the average wood failure values of Emulsion A were much higher than Emulsion B in low temperature region. Therefore the apparent bond strength of Emulsion A in this region was considered to be a strength of wood itself; so Emulsion A gave better performance.

The dramatic temperature dependence of tensile bond strength for cross-lap specimens is shown in Fig. 6 and Table 3. The tensile bond strength made a clear maximum peak around 40 C, and to our surprise, its way of variation with temperature was superimposable to that of ultimate strength of adhesive film except peaks at 30 C shown in Fig. 4. That is to say, the temperature dependency of bond strength is strongly governed by mechanical properties of the adhesive polymers.

Now we must concentrate our attention on the considerably different trends between the tensile shear test and the cross-lap tension test. In the former test a high percentage of strength was retained even as test-temperature approached −100 C. On the other hand, tensile bond strength of cross-lap joint was greatly

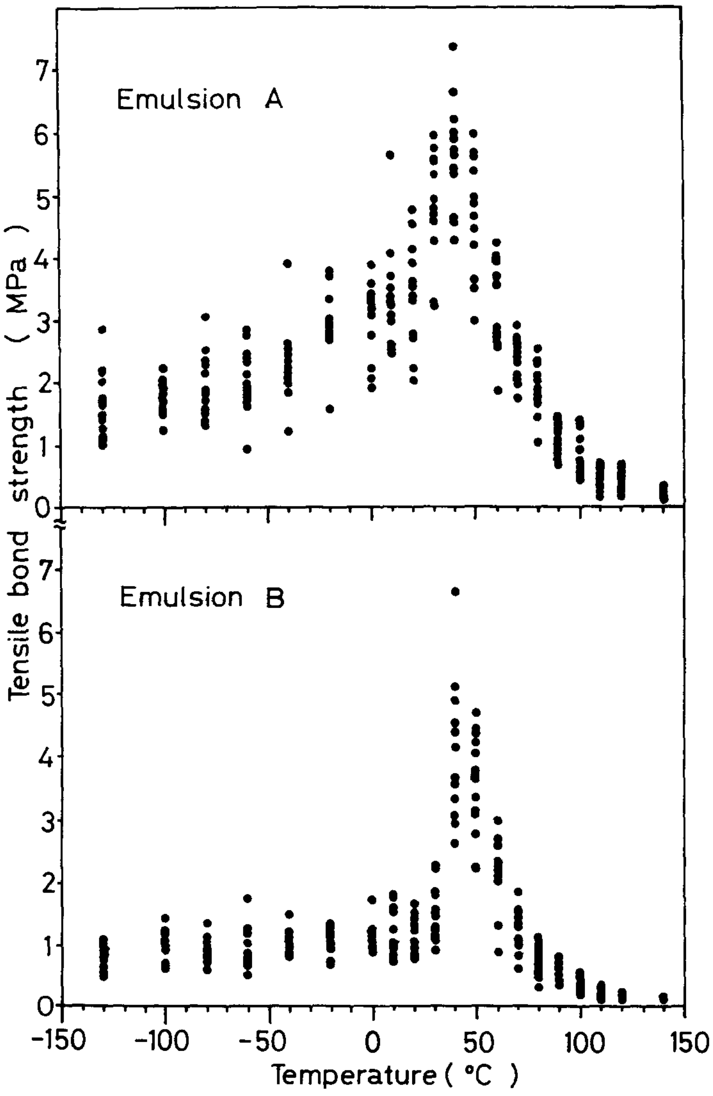

FIG. 6. Temperature dependence of tensile bond strength of cross-lap joints of two emulsion adhesives.

reduced by a slight drop of the test-temperature from 40 C. To explain this interesting difference, several experimental factors involving shrinkage of wood specimens must be taken into consideration.

When the test was performed below room temperature, the specimens were rapidly cooled in a temperature-controlled chamber before imposing any load. Therefore the internal stress, which may affect and reduce joint strength, seems to be induced in the glueline by shrinkage of wood specimens. In this respect, greater internal stress may be expected in cross-lap specimens than tensile shear specimens, since the longitudinal directions of two wood adherends are perpendicular to each other in the cross-lap. Of course we may explain the above dif-

TABLE 3. *Average bond strength and average wood failure values for cross-lap tension specimens from −130 to 140 C.*

	Emulsion A				Emulsion B			
Temp.	$\bar{X}$[1] (MPa)	S[2] (MPa)	C.V.[3]	W.F.[4] (%)	$\bar{X}$ (MPa)	S (MPa)	C.V.	W.F. (%)
−130	1.74	0.525	0.30	45.0	0.765	0.214	0.28	8.8
−100	1.82	0.251	0.14	48.8	1.00	0.243	0.24	10.0
−80	1.92	0.537	0.28	49.2	0.912	0.208	0.23	0.8
−60	2.15	0.562	0.26	27.1	1.06	0.325	0.31	4.2
−40	2.30	0.630	0.27	48.8	1.06	0.197	0.19	0
−20	2.93	0.582	0.20	57.5	1.10	0.213	0.19	0.8
0	3.01	0.637	0.21	47.1	1.15	0.202	0.18	0
10	3.38	0.876	0.26	24.6	1.18	0.331	0.28	0
20	3.42	0.863	0.25	54.2	1.30	0.245	0.19	0
30	4.85	0.889	0.18	44.2	1.50	0.431	0.29	0
40	5.67	0.879	0.16	22.5	4.11	1.12	0.27	0
50	4.69	0.930	0.20	13.4	3.74	0.749	0.20	0.4
60	3.28	0.754	0.23	0.8	2.16	0.573	0.27	0
70	2.41	0.495	0.21	0	1.26	0.341	0.27	0
80	1.85	0.655	0.35	0	0.785	0.229	0.29	0
90	1.14	0.259	0.23	0	0.585	0.101	0.17	0
100	0.817	0.313	0.38	0	0.368	0.119	0.32	0
110	0.485	0.166	0.34	0	0.271	0.131	0.48	0
120	0.475	0.169	0.36	0	0.195	0.0763	0.39	0
140	0.281	0.117	0.42	0	0.121	0.0256	0.21	0

[1], [2], [3], [4] were indicated in Table 1.

ference from this reason within extremely lower temperature region. However, a comparison of data at 30 and 40 C on cross-lap test for Emulsion B safely denies the strong influence of such internal stress in glueline. The bond strength showed a maximum of 40 C and declined to a bottom level with a slight dropping by 10 C to 30 C. This slight difference of test-temperature would not cause a large shrinkage of the adherends nor internal stress in glueline, which could be responsible for such a remarkable drop of bond strength. Further, the internal stress thus induced is hardly considered to act as the predominant factor that strongly weakens bond strength, since tensile shear strength remained at a high level together with high wood failure values even in the extremely low temperature region as mentioned previously.

Next we consider the different effects of temperature on bond strength with regard to the direction of the applied load. Ideally the applied load ought to be parallel to glueline in tensile shear test and normal to glueline in the cross-lap test. The bond strength of cross-lap joints declined markedly at lower temperatures where adhesive polymers are in the glassy state. Significantly, interfacial fracture between wood adherends and adhesives was observed with accompanying low wood failure. These results suggest a decrease in the boundary forces between wood and adhesive with a temperature decrease below 40 C. However shear bond strength did not decline and wood failure remained high in the same temperature region. Therefore it is strongly suggested that the shear bond strength in lower temperature region is derived mainly by a physical interlocking of adhesives in the wood, whose real surface is generally uneven when compared with metals. In the cross-lap test, on the other hand, there seem to be few possibilities for mechanical interlocking that would keep bond strength at a higher level, since the load is applied normal to the glueline. An interfacial fracture surface from the tension test is a replica of the wood surface.

From this concept of mechanical interlocking, shear bond strength in the glassy state should relate to the mechanical strength of the adhesive. Therefore Emulsion A with higher ultimate film strength than Emulsion B should exhibit higher shear bond strength in low temperature range. Indeed this was proved by the higher wood failure values of Emulsion A, though distinct differences were not observed in shear bond strength between the two.

A similar phenomenon suggesting the possible formation of mechanical interlocking within the surface of wood adherend was noted in a preceding work (Mizumachi et al. 1980). In that work a series of adhesives gradually differing in Tg were prepared by blending poly(vinyl chloride) and acrylonitrile-butadiene copolymer. Their ratios and the relation of their tensile shear strength at room temperature to rheological properties were investigated using both wood and aluminum adherends. In both adherends, the shear bond strength showed a maximum at the blend ratio of PVCl(50)/NBR(50). The Tg of the 50/50 blend was in the neighborhood of room temperature. As the PVCl content increased, the Tg of the adhesive increased above room temperature and the shear strength with aluminum began to decrease remarkably. No further change was observed when the blend ratio was PVCl(80)/NBR(20) with Tg of about 50 C. This trend quite resembles the present results in cross-lap test. In the case of wood adherends, on the contrary, a high bond strength was retained even in the PVCl-rich region corresponding to glassy adhesives. This different behavior seemed to arise from differences in the adherends and strongly suggested that interlocking may occur in wood having uneven surface but not in aluminum.

It is very interesting that the contribution of mechanical interlocking to bond strength seemed to change with the direction of the load applied to glueline in the present experiments. To ascertain these phenomena, it will be necessary to accumulate more data on the dependencies of bond strength on rheological properties of adhesives and direction of test-load as well as to begin microscopic study of the morphology of the interface between the glue and the adherend.

CONCLUSIONS

We evaluated the bond strength of two PVAc emulsion adhesives of differing grafting efficiency in both tensile shear and cross-lap tension joints over a broad temperature range. It was concluded that bond strength is strongly dominated by the ultimate strength of adhesive films, which in turn is dependent on rheological variations produced by temperature change—that is to say, when adhesive polymers are in rubbery state that correspond to high test-temperature, bond strength is weak due to cohesive fracture of adhesives. As adhesive polymers become hard with declining test-temperature, bond strength increases up to a maximum where the interfacial force between wood and adherend seems to be most effective. As test-temperature is lowered from that point, strength begins to fall markedly, owing to decreasing interfacial forces in the case of the cross-lap test. In case of tensile shear, on the other hand, a strong retention of bond strength is found even in the lower temperature region. These different results between two testing methods are considered to be due to whether or not mechanical interlocking of adhesives within the wood surface effectively resists the test-load imposed on the joint. In other words, when adhesive polymers are in glassy state, the interlocking acts as an effective factor for retaining shear bond strength, while in the cross-lap test it does not act effectively owing to the tensile force on the joint. This result is of importance for the practical application of wood adhesives, because the stress direction to glueline alters the effectiveness of mechanical interlocking.

Finally the amount of grafting of vinyl acetate monomer onto PVA at the time of emulsion synthesis influences bond strength and dynamic mechanical properties. It was confirmed from rheological analyses that a large amount of grafting leads to strong cohesion between the PVAc- and PVA-phases after removal of water from glueline. That is the reason why adhesive films having a large amount of grafting show higher ultimate tensile strength. Consequently such adhesives show higher bond strength over a wide temperature range than adhesives with a low amount of grafting.

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