Heat-stress resistance of hot-melt adhesives

Michael J. Ambrosini

ABSTRACT: Elevated-temperature stress resistance can be an important consideration when selecting and troubleshooting adhesives. Peel strength, shear strength, and softening point often are used as indicators of heat-stress resistance, but they can be very misleading. This paper presents an overview of a test method, developed by the Adhesion Committee of the Institute of Packaging Professionals, that compares heat-stress properties of hot-melt adhesives. It also includes a discussion on how selected variables affect the elevated-temperature stress resistance of hot-melt adhesives and examples showing how adhesive viscosity and softening point can be misleading indicators of elevated-temperature bond strength.

KEYWORDS: Adhesives, heat, hot melts, stresses, test methods.

Resistance to elevated-temperature bond failure can be an important property to consider when selecting and troubleshooting adhesives. This is particularly true when goods are stored under warm conditions or when they are shipped in warm climates and the packaging flaps have high spring-back forces.

Although test methods exist to measure adhesive peel and shear strengths, stresses on packaging bonds tend to be more complex than a simple peel or shear force. The mechanism is more of a cleavage stress, where the force is essentially perpendicular to the bond area.

Softening-point data by differential scanning calorimetry (DSC) and ring-and-ball test methods also are used as indicators of heat-stress resistance, but they too can be very misleading. The cohesive strength of an adhesive stored under warm conditions can be quite different from that of one near its melting point.

To better predict adhesive behavior in packaging applications, the Adhesion Committee of the Institute of Packaging Professionals (IoPP) has developed a test method in which heat-stress resistance can be compared on bonded samples under elevated-temperature cleavage stress. The test was designed to be performed with a minimum of cost and labor and to be versatile enough to compare stress resistance under a range of variables.

The complete procedure is published in the winter 1992 *IoPP Technical Journal*,* but an overview of the test method will be presented in this paper along with a review of how selected variables affect the heat-stress resistance of hot melts.

Procedure

The dimensions for the required test equipment are illustrated in **Figs. 1 and 2**. For optimum test reproducibility, care should be taken to construct multiple test holders and weights with similar dimensions.

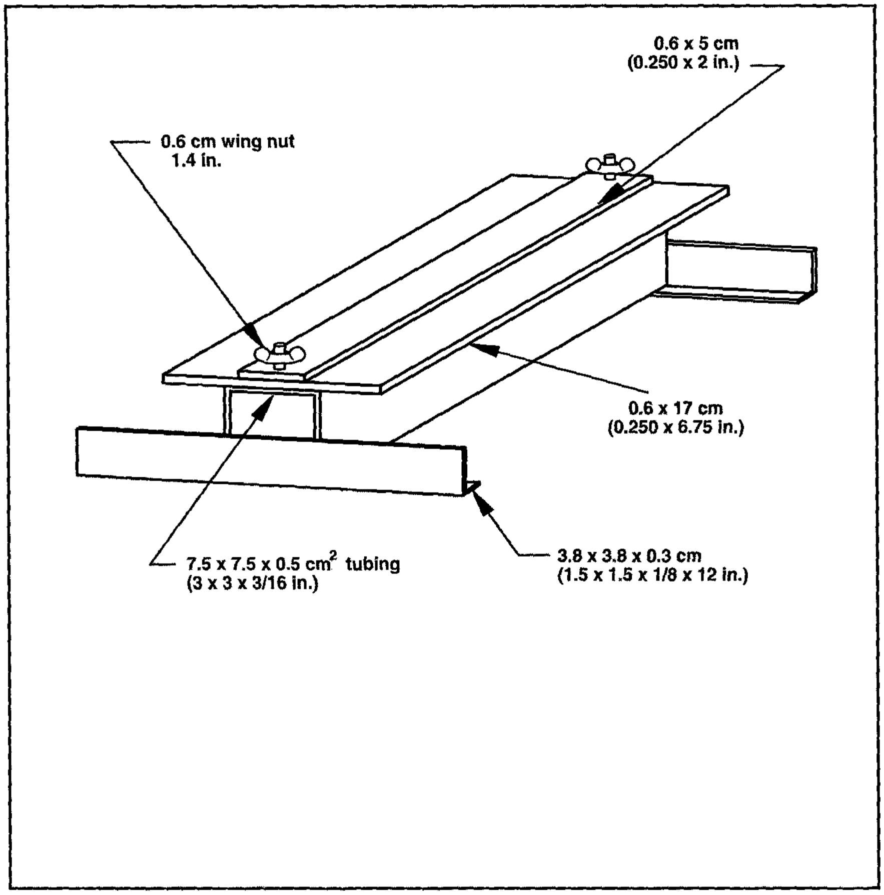
The test specimens are prepared by applying a 25-mg/cm bead (about 3 mm or 1/8-in. wide) across the center of a 5 x 8-cm piece of 1400-kPa-burst strength (200 psi) corrugated kraft board, perpendicular to the flutes, as illustrated in **Fig. 3**. The adhesive is applied at 175°C (347°F), and within two seconds open time a second piece of corrugated board stock of 5 x 14 cm is compressed onto the adhesive so that the bead is centered two centimeters from one end. The top piece also is positioned so the bead is perpendicular to the flutes.

A 3-mm hole is placed centered on the longer piece, 10 cm from the center of the bead. The specimens then are positioned onto a test holder (**Fig. 4**) so that the bead is centered four centimeters from the edge of the holder. Sample size, bead placement, sample position, and sample-holder di-

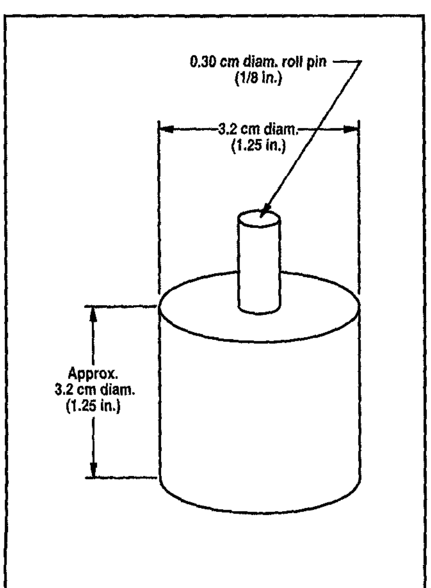
*IoPP Adhesion Committee, *IoPP Technical J.* X(1): 7–9(1992).

Ambrosini is the principal technical representative for Eastman Adhesives, P.O. Box 35369, Charlotte, NC 28235-5369.

1. Test sample holder



2. Test weight (machine length to obtain 200 g)



mensions are important for optimum test reproducibility.

The top plate is fastened down with wing nuts to secure the samples during testing. A 200-g weight is placed into the positioning hole, which is centered six centimeters from the edge of the bottom plate and 10 cm from the center of the bead. Under these conditions, the 5-cm bead is under a 60-g/cm cleavage stress force.

The entire assembly then is placed into a forced-air oven for 24 hours at a temperature controlled to ± 1°C. At least three specimens should be tested for each test in each run. If, after 24 hours, all three samples pass, new samples should be tested at a higher temperature. If the samples fail, testing should be repeated at a lower temperature. The highest temperature where three out of three samples pass, or at least 80% of the samples pass with a minimum of five test specimens, is declared the passing temperature.

The suggested starting temperature is 65–75°C (150–165°F). Once the approximate pass/fail conditions are known, the standard procedure suggests that new samples should be tested using 5°C (10°F) increments. All testing in this study, however, used increments of 5°F.

Results

To determine test repeatability, adhesive B was tested 25 times using the standard procedure (with the exception of a 5°F temperature increment). The results are plotted in **Fig. 5**. The mean passing temperature was found to be 74.5°C (166°F), with a standard deviation of 2.54°C (4.6°F). The 95% confidence limits are indicated at 69.4°C (157°F) and 79.6°C (175°F). These results indicate that adhesives with differences of less than 5°C (9°F) should not be considered significantly different.

To determine the effect of temperature on stress resistance, six specimens bonded using adhesive B were tested for 24 hours at temperatures of 74°C, 77°C, 80°C, 82°C, and 85°C using 60-g/cm stress, as noted in **Table I**. All six samples passed at 74°C (165°F), and all six samples failed at 85°C (185°F), but there is a scattering of results in between. In this case, adhesive B can be considered to pass at 74°C and fail at 77°C since 74°C is the highest temperature at which at least 80% of the samples passed.

The data in **Table II** summarize all the test results obtained in this study using adhesive B. No attempt was made to obtain even cells of information for statistical review. Several lots of adhesive were used, several board stocks were evaluated, and the testing was run over several months on three test holders in three ovens.

These data reinforce the observation that below a certain temperature (under a controlled set of conditions) any adhesive should provide consistent passes; when the temperature is raised high enough, consistent failures should occur. Between these temperatures, a range exists where both passes and failures will occur (which is typical of most experimentation).

To determine if the test results are affected by bead weight, bonds were prepared on a standard source of corrugated kraft board using adhesive B at 175°C (347°F) in 12-, 15-, 20-, 25-, and 30-mg/cm bead weights. The 25-mg/cm beads were essentially 3 mm (1/8 in.) wide. The 15-mg/cm beads were about 2.5 mm (3/32 in.) wide.

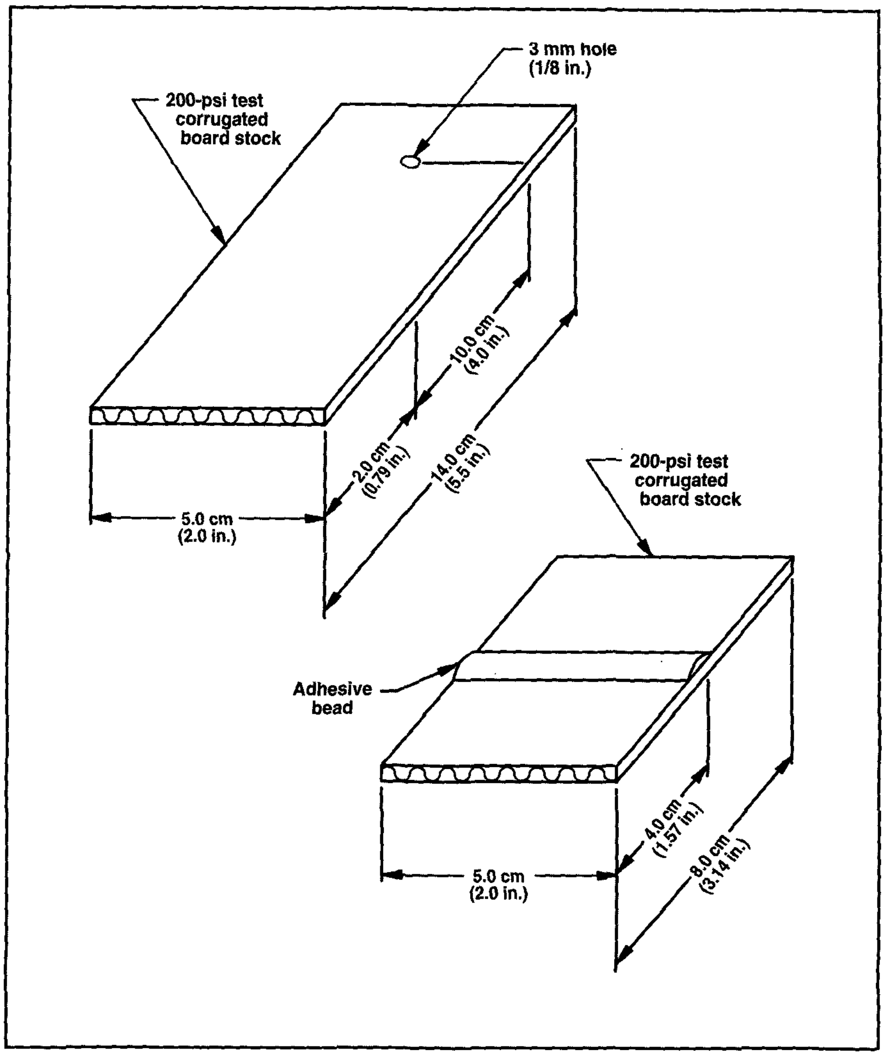
As noted in **Table III**, no significant differences resulted within this range of bead weights. This work indicates *that small variations in bead size should* not significantly affect the results. This is important if the specimens are to be prepared manually or when attempting to predict adhesive behavior in real-life situations where small fluctuations in bead size are to be expected.

Adhesive B was tested on five different 1400-kPa-burst strength (200-psi) corrugated kraft boards to determine if substrate source is a critical factor. As noted in **Table IV**, board source did not appear to significantly affect the results, but additional testing will be required before a firm conclusion can be drawn. Since all of the bond failures in this study were cohesive failures this is probably not unreasonable.

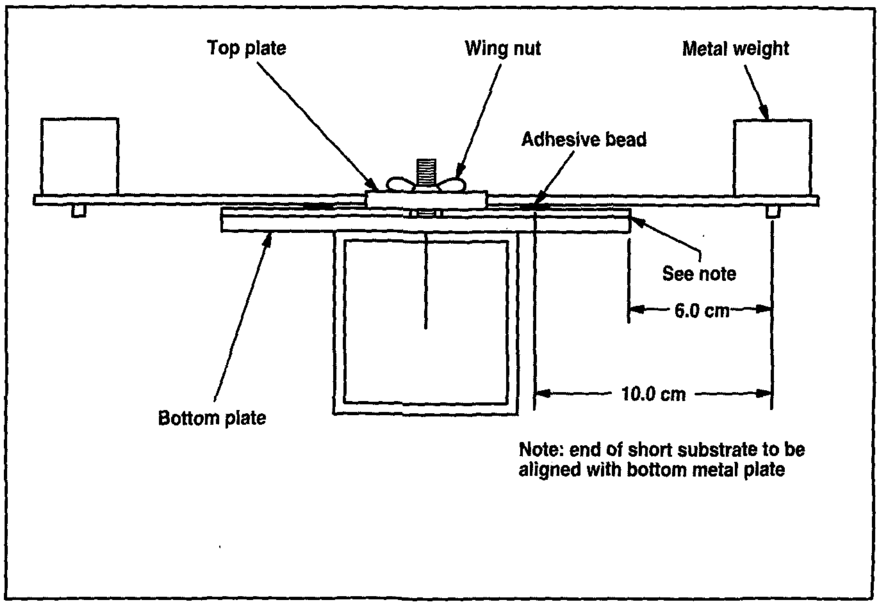
For optimum test reproducibility, it is probably best to use a single source of board stock. One restriction, however, is that the board stock must be strong enough to support the test weights without excessive bending. For consistency, all other testing in this study was prepared using a single source of board stock. Also, since the effect of sample fatigue is unknown, no samples were reused in this study.

The effect of stress force was determined by testing five different adhe-

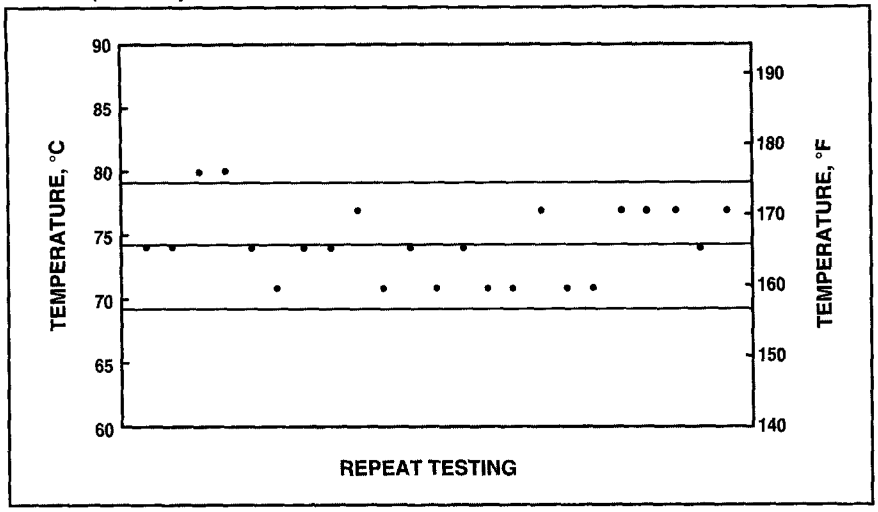
3. Test sample



4. Front view of sample holder assembled



5. Test repeatability



I. Effect of temperature on pass/fail results (adhesive B)

24 h	74°C *(165°F)*	77°C *(170°F)*	80°C *(175°F)*	82°C *(180°F)*	85°C *(185°F)*
Pass	6	4	4	1	0
Fail	0	2	2	5	6

II. Effect of temperature on all test results (adhesive B)

Temperature	*Total samples*	*Percent passing*
71°C (160°F)	57	93
74°C (165°F)	137	75
77°C (170°F)	136	66
80°C (175°F)	93	43
82°C (180°F)	28	29
85°C (185°F)	6	0

III. Effect of bead weight (adhesive B)

Bead weight, mg/cm	*Passing temperature*
12	74°C (165°F)
15 (3/32 in.)	74°C (165°F)
20	77°C (170°F)
25 (1/8 in.)	74°C (165°F)
30	74°C (165°F)

IV. Effect of board stock (adhesive B)

Board stock	*Passing temperature*
1	71°C (160°F)
2	77°C (170°F)
3	74°C (165°F)
4	71°C (160°F)
5	71°C (160°F)

sives using 100-, 150-, 200-, and 300-g weights for effective stress forces of 30, 45, 60, and 90 g/cm, respectively. The result was that heat-stress resistance was found to be critically dependent on the stress force, but the relative differences between adhesives tended to be consistent, regardless of the stress.

Adhesives A and B are polyethylene based, adhesive C is based on EVA (ethylene vinyl acetate), and adhesives D and E are based on amorphous polyolefins (**Fig. 6**)

Without defining the stress force, the values by themselves tend to be meaningless, but they can be quite useful when comparisons are made on a relative basis. For example, values will change depending on the particular stress force, but adhesive B should provide improved stress resistance as compared to adhesive C, D, or E under any cleavage stress.

If heat-stress problems are occurring with a particular adhesive, a product with a higher test result should be evaluated. Products with lower ratings should not be considered.

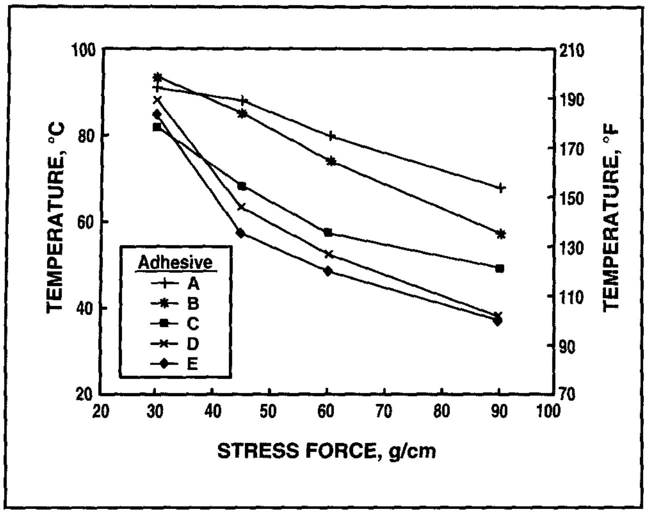
The IoPP procedure states that the samples should be held for 24 hours at temperature. To determine if a shorter time can be used, samples bonded with adhesives B and D were tested at various temperatures, and the maximum time the samples could withstand the 60-g/cm stress force was recorded for each temperature.

As noted in **Fig. 7**, the results for adhesive B did not stabilize until after about 24 h. The results for adhesive D did not stabilize for about 72 h, but the results beyond 24 h may not be statistically different. Adhesives A, C, and E were tested only at and beyond 24 h, but with similar results. It appears that the more crystalline products may stabilize faster than the amorphous products, but statistically there may not be a significant difference in test results beyond 24 hours for any of these products.

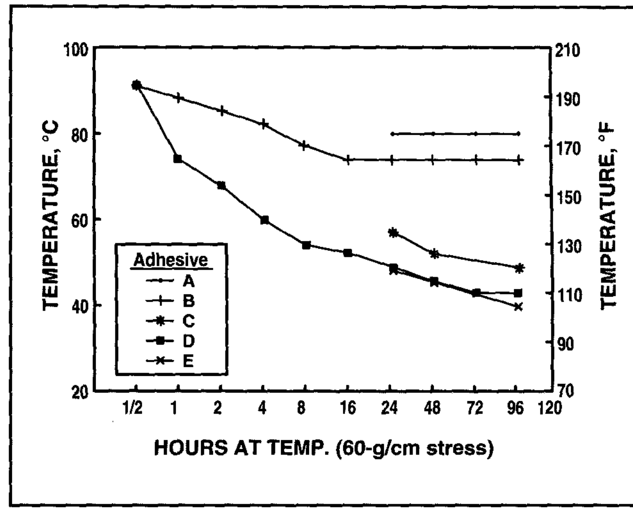
Although the results are dependent to a degree on the exposure time, the relative ranking of adhesives remained consistent regardless of the exposure time. There may be some advantages in using a shorter exposure time to estimate an approximate passing temperature or to quickly identify relative differences between products, but a 24-h exposure is probably best for long-term comparisons.

Softening point often is thought to be an indicator of elevated-temperature bond strength, with the belief that an adhesive with a higher softening point should have improved elevated-temperature stress resistance as compared with one having a lower

6. Effect of stress force



7. Effect of time vs. temperature



V. Effect of softening point

Adhesive	*IoPP (60 g/cm)*	*Ring-and-ball*	*DSC*
A	80°C (175°F)	105°C (221°F)	93°C
B	74°C (165°F)	109°C (228°F)	82°C, 100°C, 107°C
C	57°C (135°F)	107°C (225°F)	91°C, 98°C
D	52°C (125°F)	117°C (243°F)	91°C
E	49°C (120°F)	145°C (293°F)	86°C, 144°C

VI. Effect of viscosity

Adhesive	*IoPP 60-g/cm passing temp.*	*Viscosity, cp 177°C (350°F)*
A	80°C (175°F)	21,000
B	74°C (165°F)	850
C	57°C (135°F)	900
D	52°C (125°F)	1,300
E	49°C (120°F)	2.700

softening point. This may be true in some cases, but, as noted in **Table V**, adhesives D and E have much higher ring-and-ball softening points vs. adhesives A and B, but they also have significantly reduced heat-stress resistance.

Viscosity is another physical property often considered when predicting elevated-temperature bond strength. In **Table VI**, we see how adhesives D and E have higher viscosities than B or C, yet they have significantly reduced heat-stress potential as compared to adhesives B and C. Viscosity may be one factor that influences elevated-temperature bond strength, but clearly there are other factors that may be more significant.

Summary

There are probably many factors that determine the heat-stress resistance of a hot-melt adhesive, but measuring each one and attempting to determine all the interactions can be a complex, costly, and time consuming exercise.

The test method proposed by the IoPP Adhesion Committee appears to be a relatively reliable and practical means of comparing adhesive behavior with a minimum of cost and labor. Although the exact bond stress is rarely known, and the test results are dependent on stress force, through relative comparisons this test method can provide useful information about the elevated-temperature stress resistance of hot-melt adhesives, particularly when selecting, comparing, or troubleshooting hot melts.

Received for review March 23, 1993.

Accepted April 5, 1993.

Presented at the TAPPI 1993 Hot Melt Symposium.