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Data Analysis on Adhesive Property Testing Methods of Hot-melt Pressure-sensitive Adhesive with Styrene Thermoplastic Elastomer

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Abstract. In this study, the adhesive property testing methods of hot-melt pressure-sensitive adhesive containing styrene thermoplastic elastomeric used in Non-woven material surface bond were studied. The coating thickness, sticking time before test, aging time, peeling rate, angle of initial adhesion test and material of permanent adhesion test were analyzed, which refers to the parameters in the process of test samples preparation. The experiment results showed that the HDPE test plate is better than the traditional steel test plate for the measurement of the HMPSA permanent adhesion.

Introduction

Hot-melt pressure-sensitive adhesive(HMPSA) is a kind of adhesive with both hot-melt adhesive and pressure-sensitive adhesive properties, which consists of thermoplastic elastomer as the main material, adding tackifier, plasticizer, antioxidant and other additives[1,2]. This kind of adhesive coatting on a film under the molten state can be rapidly bonding after applying light pressure when it is cooling down and hardening, at the same time it can more easily be stripped without pollution on bonding surface. Its main application fields contain packaging, label, Non-woven material, special adhesion and other fields.

HMPSA with styrene thermoplastic elastomer was composed of styrene block copolymer as main material and was applied to the surface bond of non-woven materials [3,4]. Its adhesion property is one of the most important performances, and the reliability of its adhesion to non-woven fabrics is significant. There are three main test methods: initial adhesion test, peeling strength and permanent adhesion test. However, there are two deficiencies in the test method of HMPSA: first, the initial viscosity of the adhesive can be tested by the slope ball method, but it is not easy to quantify when its value is too small. Second, the test method of peeling strength is not precise enough, and the test results of this kind of adhesive are quite different in different conditions.

The national standard test criterion is designed mainly for adhesive tape products. However, for HMPSA products with styrene thermoplastic elastomer for Non-woven material, this method will be subjected to coating thickness, storage time, interfacial type and roughness, temperature and other lab conditions during the test process. For any carelessness

during the test, the result would be a severe deviation. In addition, if the parameters are not determined and the experimental conditions are not strictly controlled, the test results are often not comparable.

Therefore, it is necessary to establish the corresponding adhesion performance test method for the storage environment and use of hot-melt pressure-sensitive adhesive with styrene thermoplastic elastomer.

Test Section

Testing Materials. HMPSA samples (#7701 series, #7702 series), Ligao Adhesive (Boxing) Co., Ltd..

Testing Instruments. Non-continuous hot-melt adhesive coating machine, TX2012-1, Ligao Adhesive (Boxing) Co., Ltd.. Electric blast drying oven, 101-DA, Tianjin Test Instrument Co., Ltd.. Initial adhesion tester, CZY-G, Jinan Languang Mechanical and Electrical Technology Co., Ltd.. Permanent adhesion Tester, CZY-6S, Jinan Languang Mechanical and Electrical Technology Co., Ltd.. Intelligent Electronic Stripping Tester, XLW-100N, Jinan Languang Mechanical and Electrical Technology Co., Ltd.. Constant Temperature and Humidity Box, HS/YH-150, Chongqing Yongsheng Experiment Instrument Factory.

Experimental methods. The coating equipment is a self-made hot-melt adhesive coating machine. HMPSA sample should be coated on the PET film in a hot stage. The coating thickness is controlled by the grip, the coating temperature is appropriate when the sample has a better flow condition, and the sample should be covered by the anti-adhesive layer after cooling and stored for measure.

Initial adhesion test is conducted according to GB/T4851-1998 criterion, permanent adhesion test is conducted according to GB/T4851-1998 criterion, 180 ℃ melt viscosity test is conducted according to HG/T 3660- 1999 criterion, peeling strength test is conducted according to GB/T2792- 1998 criterion and aging test is conducted according to GB / T3512-2001 criterion.

Results and discussion

Coating thickness of HMPSA samples. The hot melt pressure sensitive adhesive product # 7702 was obtained and melted. Its test sample was prepared in the coating machine with different thickness setting. Preheating 10min to prevent deformation caused by heat expansion and cold contraction, upper and bottom roller temperature of the coating machine was keeping at 100 ℃, and the sample temperature was keeping at 150 ℃. After storing for the same time period, the samples were cut into 25mm test strip, adhered to the test paste board. Each sample with different thickness 20μm, 30μm, 50μm, 70μm, 100μm was measured three times for its peeling strength, and the mean value was taken as the result.

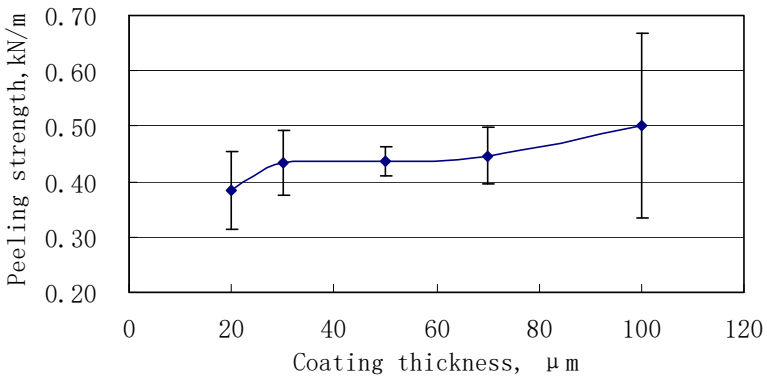

Fig.1 Influence of coating thickness to peeling strength

As shown in Fig.1, the samples with coating thickness less than 30μm and more than 70μm got significantly different peeling strength test results. The data of peeling strength is much lower (<0.40 kN/m) or higher (>0.48 kN/m) with quite large standard deviation value (> 0.07). This is mainly because occasional factors will affect the test results when the thickness is too small and the HMPSA material will creep when the thickness is too large. According to the peeling strength variation tendency and the standard deviation value (0.025), 50 ± 5μm coating thickness can be chosen appropriately.

The sticking time on the test board before 180° Peeling Strength test. Eight #7701 products were prepared for melting by the coating machine and coating on single-sided silicon treatment of the release film. The samples were covered by the release paper and stored at room temperature for 24h. Then the peel strength was measured after the samples were affixed on the metal paste board of the electronic stripping machine, respectively, for 30min and 2h. The following data was obtained (See Table1).

Table 1 The sticking time on the test board before 180° Peeling Strength test

Samples	#7701-1	#7701-2	#7701-3	#7701-4	#7701-5	#7701-6	#7701-7	#7701-8
30min test of peeling strength(kN/m)	0.41	0.92	0.70	0.36	0.43	0.43	0.56	0.58
2h test of peeling strength(kN/m)	0.89	0.91	1.31	0.43	0.94	1.11	0.84	0.89

At the beginning, the adhesion of the specimen to the metal test plate is small, this is mainly because the force generated by the molecular chain and the metal plate in a short period of time is relatively small, however, over time, more bonding "point" will produce between the hot-melt adhesive and the test plate, thereby, the cohesive force gradually increased.

Taking another type of hot-melt adhesive product #7702 for example, a long time comparison test was conducted for the same sample, which can also explain the trend demonstrated above. In this case, the sample thickness is 50μm.

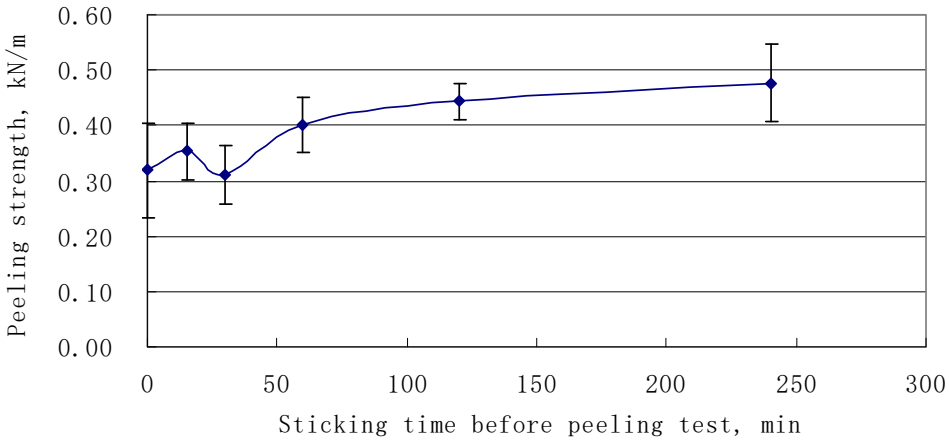

Fig.2 Influence of sticking time before test to peeling strength

As can clearly be seen in Fig.2, at the early stage after the sample being taped on the metal paste plate, the standard deviation data of peeling strength was quite large (0.09). The total data within the first one hour fluctuated greatly (from 0.31 kN/m to 0.35 kN/m). The peeling strength is stable in around 0.44 kN/m and the standard deviation data is small (0.03) when the test specimen is applied to the metal paste plate for 2h. However, the peeling strength increased significantly (＞0.48kN/m) when the test time is more than 4h and, even more, it was 0.80 kN/m at 24h which has the standard deviation 0.13. This is mainly because HMPSA forms a more solid bond with metal plate at this time, which is due to the metal plate surface characteristics.

Under the test conditions, test is more appropriate, then peel strength is more stable; time more than 4h, which is due to the metal plate surface characteristics of the decision, this time heat Melt and metal plate to form a more solid bond, and the adhesive in the application and the material from the type of bonding will not appear such a big break. The product #7701 series expressed more significant change than #7702 series product during the sticking time.

The peeling rate during the test of 180° peeling strength. The current peeling strength test adopted the test standard GB/2792-1998 criterion, using BLD-S-type electronic universal stripping machine for testing. the sample was cut into 25×l00mm strip, attached to the clean stainless steel plate. The samples were allowed to stand for 20 min and then tested at a constant speed of 300 mm/min. Each group of test was tested for at least three samples. Test conditions: temperature 23±2 °C, relative humidity 65±5 %. In order to discuss the influence of different peeling rate, three groups of experiment were prepared.

Two hot-melt adhesive products, #7701-128 and #7702-100F, were used to prepare samples for the peeling strength test. BLD-S electronic universal stripping machine was set different peeling rate (100mm/min, 200mm/min and 300mm/min respectively). The test results were as follows (See Table 2).

Table 2 The peeling rate during the test of 180° peeling strength

Peeling rate (mm/min)	100		200		300	
Samples No.	7701-128	7702-100F	7701-128	7702-100F	7701-128	7702-100F
Peeling strength(kN/m)	1.54	0.14	1.55	0.13	Backing break	0.18
Peeling power range(N)	22.72-28.52	6.18-8.07	23.19-29.49	4.75-8.42	——	0.16-22.40

Based on the specific data, when the peeling rate of 300mm/min is conducted, the test deviation of both two kinds of HMPSA products is comparatively large. For some kind of samples with large peel strength, the specimens will be broken because the local bond strength of the samples is too large. For the peeling rate 100 mm/min, the test process appears peak value and then drop value dramatically. Therefore, the medium peeling rate is beneficial to the uniform collection of peeling strength, and 200 mm/min peeling rate is used for the follow-up test.

Effect of storage time on peeling strength after HMPSA Coating. After the HMPSA being heated, a 50 mm thick sample was prepared on a coating machine and was set at room temperature. During the placement process, the hot melt is subjected to a transition from a flow state to a semi-flow state and then to a solid state. Its internal force is also changing, so the appropriate time to determine the peeling strength should be selected. Consequently, not only the peeling strength stably reflects the value of the HMPSA cohesion, as well the testing time is saved greatly.

Three #7701 samples were tested, respectively, at different storage time under condition of temperature 23±2 °C, relative humidity 65±5 %. The samples were pasted on the metal test board for 2h before the test. The results are as follows (See Table 3).

Table 3 Effect of storage time on peeling strength after HMPSA Coating.

Storage time	Peeling strength (kN/m)		
	#7701-1sample	#7701-2 sample	#7701-3 sample
10min	0.08 (adhesive material drawing)	0.11 (adhesive material drawing)	0.12 (adhesive material drawing)
1h	0.31	0.49	0.55
6h	1.17	1.22	1.31
24h	1.30	1.39	1.44
72h	1.35	1.37	1.45
300h	1.29	1.30	1.37

During the initial 1-6 hours, adhesive material drawing phenomenon occurs occasionally. This is mainly because HMPSA is In the semi-flow state after melting, the viscosity of the system is much lower, the interaction force between the molecular chains is weak, and the cohesion is less than the adhesion force. When the temperature is completely reduced to room temperature after 24h storage, in the stage of 24 h to 300 h, cohesion tends to be smooth (1.29-1.35 kN/m, 1.30 -1.37 kN/m and 1.37-1.45 kN/m, respectively, in each group of experiment) the HMPSA is completely solid, its cohesion increases, a certain creep occurs with the HMPSA, and the molecular chain may have shifted or adjusted. After the sample is prepared for at least 24h, the adhesion between the sample and the test plate is greater than that between the samples. Then the peeling strength of the sample during the peeling process maximumlly shows the interaction force between the molecules. Therefore, 24h storage time is necessary serving as a test standard.

Initial adhesion and permanent adhesion property test. HMPSA sample is test according to GB 4852-84 pressure sensitive adhesive tape initial adhesion test standard (bevel roller method). The greater the value of the test sample sticking to the ball shows that the surface bond strength is stronger, namely, the initial viscosity is comparatively strong(See

Table 4).

Table 4 Initial adhesion and permanent adhesion property test

Samples	#7701-1	#7701-1	#7701-1	#7702-1	#7702-2	#7702-1	#7702-2
Specimen thickness(μm)	50	60	65	50	50	55	55
Initial adhesion(inclination 30°)	—	—	20#	—	27#	27#	33#
Initial adhesion(inclination 15°)	16#	18#	24#	36#	40#	42#	48#

The results show that less than 20 # steel balls can not be stuck for all the samples with the inclination on 30° because the rolling speed of the ball is comparatively fast. The thickness of the specimen has a significant effect on the initial adhesion. The initial adhesion will increase with the increasement of the thickness of the specimen. The #7701 series product, as a product with more weak initial adhesion property than that of the #7702 series, can not be measured about its initial adhesion value when its thickness is less than 65μm. So in this case, the measured value can not reflect the true value of different samples. When the inclination was adjusted to 15° to measure the initial adhesion, all the measured samples' values were obtained because the speed of the ball slows down. The initial adhesion value was larger than that of inclination on 30° and properly reflected the contrast of different initial adhesions of each sample.

Three samples of #7701 were used for permanent adhesion test using steel test plate and HDPE test plate respectively (See Table5).

Table 5 Initial adhesion and permanent adhesion property test

Specimen No.		#7701-1	#7701-2	#7701-3
Permanent adhesion for different test plate	Steel plate	＞72h，interfacial debonding failure	＞ 72h， interfacial debonding failure	＞ 72h， interfacial debonding failure
	HDPE plate	＞2 weeks，cohesive failure	＞2 weeks，cohesive failure	＞2 weeks，cohesive failure

In the case of permanent adhesion test, when the steel plate was used, the three specimens were interfacial damaged with a short sticking time, with no cohesive failure. Therefore it can not really reflect the cohesion difference between the specimens. However, there was a distinct difference when the HDPE plate, which has a melting point of about 130 °C, a relative density of 0.941 to 0.960 and a surface roughness of 50±25 nm, is used. The damage of adhesives is cohesion failure, and the permanent adhesion time is more than 2 weeks. This is mainly because HDPE plate surface property determines it is more easily bonded by the adhesive, the surface adhesion increases significantly, so the adhesive strength is larger than cohesion. The HDPE plate is suitable for permanent adhesion comparison between different samples.

Conclusion

For hot-melt pressure-sensitive adhesive containing styrene thermoplastic elastomer used in Non-woven material surface bond, 50±5 μm coating thickness can be chosen appropriately in the test process, 2h after the preparation of the sample on the test plate in the test condition is

appropriate to obtain stable peeling strength results, 200mm/ min peeling rate is conducive to data uniformly collection of the peeling strength. Samples should be stored at least 24h after the preparation, when the material adhesion is greater than cohesion, which truly reflects the adhesion performance of specimens. Adjusting the inclination to 15° to measure the initial adhesion, the measured value can reflect the comparison between different samples. The HDPE test plate is better than the traditional steel test plate for the measurement of the HMPSA permanent adhesion.

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