Research on processing technology and properties oflow-viscosity adhesive sheets

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Abstract: As a crucial basic material in the manufacturing of multilayer printed circuit boards (PCB), prepreg, despite its relatively thin thickness, demands high standards for surface flatness, mechanical properties, and surface tackiness. Research indicates that tension can affect the average thickness, while tension, vehicle speed, and adhesive viscosity all influence the tackiness of the material. Through DOE experiments to optimize process parameters, the material exhibits excellent surface flatness (with a thickness standard deviation of 0.0071) and an extremely low tacky area ratio (10%) when subjected to a tension of 25 N, a vehicle speed of 13 m/min, and an adhesive viscosity of 410 cps. SEM examination reveals no microcracks in the material, with its tensile strength reaching 10.68 MPa, an increase of over 35%, and its elongation at break reaching 74.1%, an increase of over 33%.

Key words: adhesive sheet; glue viscosity; tensile strength; elongation at break

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0 Introduction

With the development of electronic devices towards high performance and miniaturization, the demand for multi-layer printed circuit boards (PCBs) is growing. As a key material for interlayer bonding in PCBs, the performance of prepreg directly affects the reliability of PCBs. Traditional prepreg materials are usually composed of resin-impregnated glass fiber cloth, which needs to be fully cured during the subsequent lamination process to achieve interlayer bonding. The dielectric loss of this material is limited by raw materials and processing techniques, making it difficult to achieve a value lower than 0.0015. PTFE materials have attracted increasing attention due to their extremely low loss (theoretical loss value of 0.0009). However, PTFE has self-lubricating properties and lacks viscosity, making it difficult to achieve bonding performance directly using this material.

Wang and his team used sintered fusible polytetrafluoroethylene as the matrix resin to prepare fluororesin flexible copper clad laminates. In practical applications, the thickness of the bonding sheet material is usually required to be extremely thin (generally less than 120 μm), but the surface flatness is highly required. The use of two or more layers of multi-layer bonding sheets is also common. However, misalignment, bubbles, and overlapping wrinkles between bonding sheets during stacking can seriously affect the processing quality of PCBs. Therefore, in the research work on bonding sheets, the evaluation of the mechanical properties (tensile strength and elongation at break) and tackiness of the material is also crucial. The use of impregnation process to produce bonding sheets is prone to adhesion failure due to high tackiness during cutting, inspection, packaging, and transportation. Moreover, during the multi-layer lamination process by users, problems such as delamination and board bursting due to low mechanical properties are easily encountered, affecting the final product quality.

This study systematically analyzes the impact of key parameters in the impregnation process on the microstructure,

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surface flatness, viscosity, tensile strength, and elongation at break of adhesive sheets. Based on this analysis, an optimization scheme is proposed to achieve the preparation of adhesive sheet materials with low viscosity, high flatness, high tensile strength, and high elongation at break.

1 Experimental part

1.1 Main materials and equipment

The raw materials used mainly include: PTFE film (76 μm), Tianjin Plastics Research Institute; hydrocarbon resin: polybutadiene, Beijing Yanshan Petrochemical Co., Ltd.; crosslinking agent: dicumyl peroxide (DCP), commercially available; and additives such as silane coupling agent and co-crosslinking agent, commercially available.

The equipment used mainly includes: a precision impregnation machine (self-developed, working width 1000 mm, maximum speed 30 m/min, tension (5~50 N)), a high-temperature oven (maximum temperature 500 ℃), and Shanghai Boxun.

1.2 Process parameter design

The experiment selected tension (10~40 N), vehicle speed (5~15 m/min), and glue viscosity (250~450) as variables, and employed the DOE experimental design method to investigate the effects of tension, vehicle speed, and glue viscosity on the mean thickness, thickness uniformity, and surface adhesion area ratio of the bonded sheet. The process parameter design is shown in Table 1.

1.3 Performance testing and characterization

The performance tests of samples prepared with different process parameters are shown in Table 2.

Surface flatness: The thickness of the adhesive sheet is measured using an X-ray thickness detection system, with 35 points tested per meter within a width of 1,000 mm. The mean value and standard deviation are calculated, and the standard deviation represents its surface flatness.

Glue viscosity: characterized by Brookfield LVDVS+ viscometer.

Adhesion Test: According to the test method described in IPC-TM-650 2.4.1, firmly press a 50 mm long pressure-sensitive adhesive tape onto the surface of the test sample, squeezing out any trapped air. The time interval between applying and removing the tape is 30 seconds. Visually inspect the tape and the test area to check if any part of the test material has peeled off from the test sample. If there is any peeling, record the area ratio (%) of the peeled-off part. If there is no peeling, record it as 0.

Mechanical properties: According to GB/T 1040.2-2006, the tensile strength and elongation at break of the film are tested using a universal tensile machine, with 5A standard specimens selected.

Microscopic morphology: The microscopic surface roughness of the bonded sheet was observed using a SUPPRA 55VP field emission scanning electron microscope from Zeiss, Germany.

2 Analysis of the influence of process parameters on the performance of adhesive sheets

2.1 Microscopic morphology analysis

Figure 1 shows the surface micromorphology of bonded sheets produced with different process parameters. Figure 1(a) represents sample 7, Figure 1(b) represents sample 11, Figure 1(c) represents sample 9, and Figure 1(d) represents sample 2. Scanning electron microscopy reveals that process parameters have a significant impact on surface quality. When the tension is low, the surface appears uneven and rough, as shown in Figure 1(a). When the tension is high, microcracks appear on the material surface due to excessive stretching, as shown in Figure 1(b). As the vehicle speed increases, both the size and number of microcracks significantly increase, as shown in Figure 1(c). As the viscosity decreases, the depth of microcracks tends to increase, as shown in Figure 1(d).

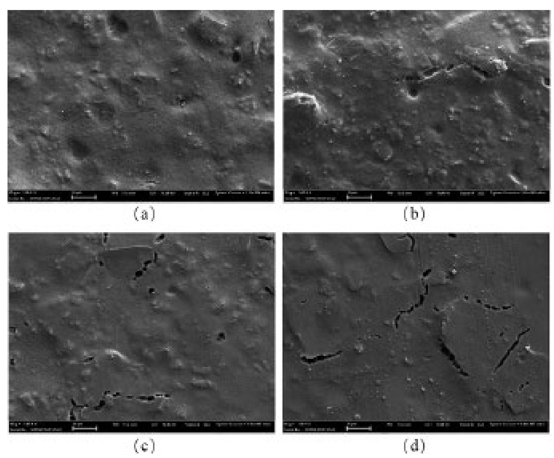

Figure 1 Surface micro-morphology of the sample

Table 1 Process parameter design table

Serial number	Tension/N	Vehicle speed / (m·min⁻¹)	Glue viscosity/cps	Serial number	Zhang Li	Speed	Glue viscosity/cps
1	25	10	350	7	10	5	450
2	40	15	250	8	10	15	450
3	10	15	250	9	40	15	450
4	25	10	350	10	10	5	250
5	25	10	350	11	40	5	450
6	40	5	250	/	/	/	/

Table 2 Thickness and viscosity tests of samples prepared with different process parameters

Serial Number	Mean Thickness/mm	Surface Flatness/ mm	Viscous Area Ratio/%	Serial Number	Mean thickness (in mm)	Surface Flatness /mm	Viscous Area Ratio/%
1	0.097	0.002	15	7	0.097	0.01	10
2	0.082	0.005	40	8	0.099	0.009	10
3	0.082	0.005	20	9	0.091	0.009	10
4	0.097	0.003	15	10	0.098	0.009	50
5	0.099	0.002	15	11	0.118	0.01	10
6	0.084	0.007	50	/	/	/	/

2.2 The influence of process parameters on the mean thickness and uniformity of adhesive sheet materials

Tension, vehicle speed, and glue viscosity are key parameters in the impregnation process, directly affecting the average thickness and uniformity of the material, and indirectly influencing key indicators such as the material's mechanical properties. Among them, tension indirectly affects the leveling of the glue by influencing the degree of deformation of the material. Moderate tension ensures the material is flat and provides a stable coating surface for the glue. Vehicle speed directly affects the leveling time of the glue, thereby influencing the amount of glue applied and ultimately the thickness and uniformity of the material. Glue viscosity directly affects the viscosity and reactivity of the resin, thereby influencing the flowability of the glue during the impregnation process.

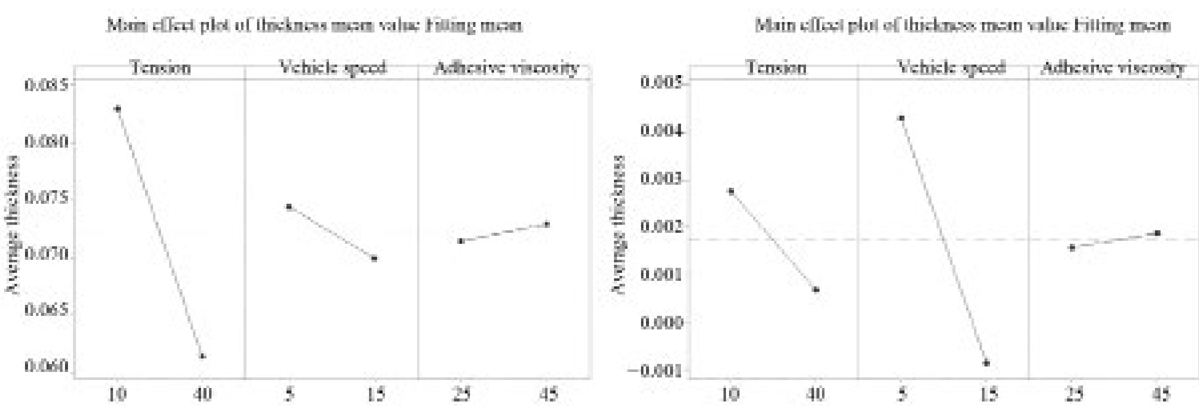

Figure 2 Main effect plot of process parameters on thickness mean and uniformity

The experimental results indicate that tension has a significant impact on the mean thickness (see the slope relationship between tension and mean thickness in the main effect plot on the left of Figure 2), but its effect on thickness uniformity is slightly less pronounced (see the right panel of Figure 2)

The relationship between tension and the slope of the mean thickness uniformity in the main effect diagram of thickness uniformity. Too low tension can cause material relaxation. When the tension is low (10 N), the material is prone to wave-like deformation in local micro-regions, making it difficult for the glue to flow evenly and level off after gluing, resulting in microscopic unevenness in local areas, as shown in Figure 1(a). As the tension increases (25 N), it is beneficial to maintain material stability, with the glue spreading evenly and good thickness uniformity. However, excessively high tension

(45 N) can cause microcracks in the material due to excessive stretching, as shown in Figure 1(b), affecting the mechanical strength of the bonded sheet.

Vehicle speed has a certain impact on both the mean thickness and thickness uniformity (see the slope relationship between vehicle speed and mean thickness in the main effect plot of mean thickness on the left in Figure 2, and the slope relationship between vehicle speed and mean thickness uniformity in the main effect plot of mean thickness uniformity on the right in Figure 2). Too fast a vehicle speed may cause the adhesive to cure before it has fully leveled, resulting in surface defects; too slow a speed may lead to excessive penetration of the adhesive, causing localized thickening of the material. Optimizing the vehicle speed can balance production efficiency and surface quality. When the vehicle speed is low (5 m/min), the adhesive has sufficient time to level, but too low a speed can cause the adhesive to dry and cure locally, which is not conducive to surface flatness. As the vehicle speed increases, the leveling and curing speeds of the adhesive reach a balance, resulting in better surface flatness. When the vehicle speed is too high, the adhesive has insufficient time to level, leading to local "orange peel cracking" phenomena, as shown in Figure 1(c).

The viscosity of the glue solution has a certain impact on both the mean thickness and the uniformity of thickness (see the slope relationship between glue solution viscosity and mean thickness in the left main effect plot of mean thickness in Figure 2, and the slope relationship between glue solution viscosity and mean uniformity of thickness in the right main effect plot of mean uniformity of thickness in Figure 2). Too high a viscosity leads to poor fluidity of the glue solution, resulting in a significantly thicker material with uneven thickness. As the viscosity decreases, the fluidity of the glue solution increases, and the uniformity of thickness is improved to some extent.

2.3 The influence of process parameters on the viscosity of adhesive sheet materials

In general, the glue solution is the factor that has the greatest impact on the viscosity of materials, especially as changes in the viscosity of the glue solution can affect the viscosity and reaction activity of the resin, thereby influencing the viscosity of the material. Through data analysis of the DOE experimental design, it is found that tension and vehicle speed also have an impact on the viscosity of materials (see the slope relationship between tension, vehicle speed, and viscosity mean values in the left viscosity main effect plot of Figure 3). It is worth noting that there is also a certain interaction between tension and vehicle speed (see the extension lines of tension and vehicle speed intersecting in the upper left position of the viscosity interaction plot in Figure 3).

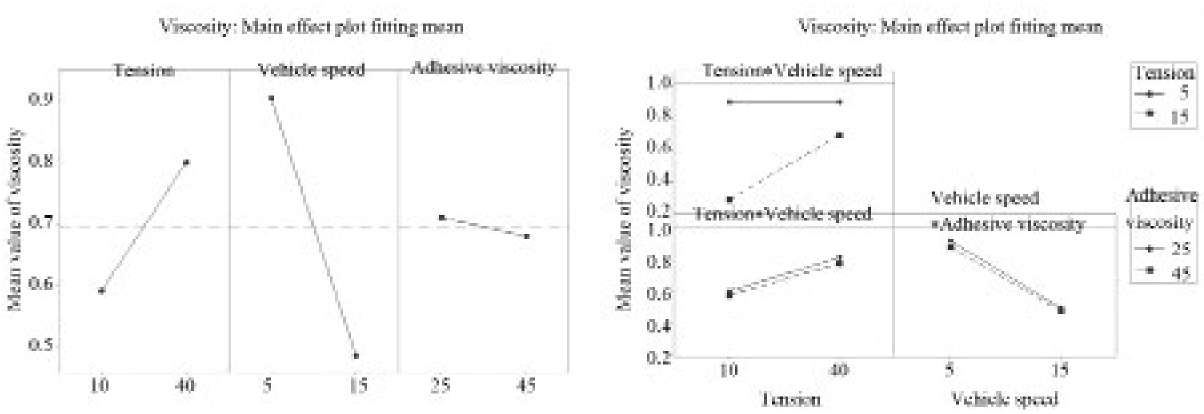

Figure 3 Main effect and interaction plots of process parameters on viscosity

The experimental results indicate that, compared to tension, the influence of vehicle speed on material viscosity is slightly more significant. When the vehicle speed is low, it is easy to cause excessive resin penetration, thereby increasing the material viscosity. When the viscosity of the glue solution is low, the surface viscosity of the material after impregnation is relatively high. As the viscosity of the glue solution further increases, pre-curing phenomena appear on the resin surface, and local cracking becomes more pronounced, as shown in Figure 1(d). This is not conducive to subsequent processing and use. Special attention should be paid to process matching adjustments when optimizing the tension and vehicle speed process parameters.

Based on the characterization and analysis results presented in Figures 1, 2, and 3, by controlling tension, vehicle speed, and glue viscosity, it is possible to alter the rheological properties of the resin. This, in turn, allows for the simultaneous optimization of three key indicators: thickness average, uniformity, and surface viscosity, while improving the surface quality of the material.

3 Optimization process and perform ance verification of low-viscosity adhes ive sheets

Based on the comprehensive influence of tension, vehicle speed, and glue viscosity on material thickness and surface tackiness, a DOE response optimizer utilizing statistical analysis tools was employed to identify the matching parameters of the three factors. By controlling tension, vehicle speed, and glue viscosity, the rheological properties of the resin were altered to enhance the uniformity of material thickness and surface tackiness. The following simulation results were obtained through the response optimizer, as illustrated in Figure 4. When the tension was 25 N, the vehicle speed was 13 m/min, and the glue viscosity was 410 cps, the simulation results met the expected targets. The expected results yielded a bonding sheet material with a surface tackiness of 12.07%, thickness uniformity (standard deviation) of 0.0069 mm, and a thickness mean value of 0.0956 mm, achieving joint optimization of material surface tackiness, thickness uniformity, and thickness mean value.

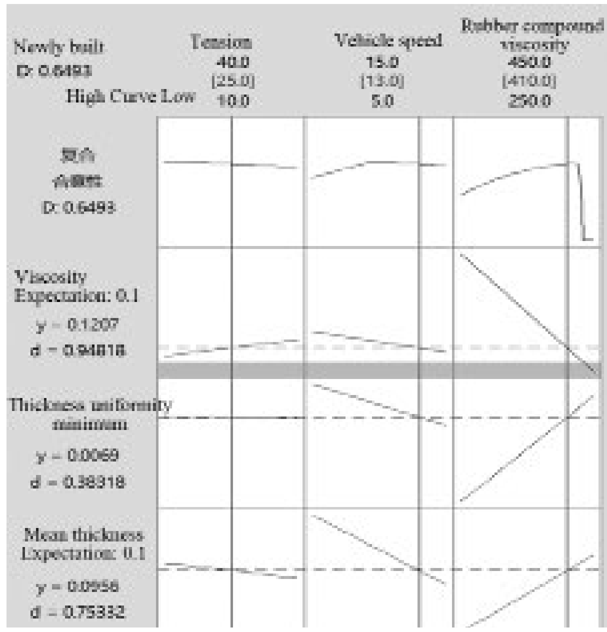

Figure 4 DOE response optimizer

Using the aforementioned parameters, low-viscosity adhesive sheet materials were continuously produced, and tests were conducted on their micro-morphology, average thickness, thickness uniformity, surface viscosity, tensile strength, and elongation at break. The test results for micro-morphology are shown in Figure 5, and the results for other tests are presented in Table 3. The process validation product is designated as PP, while others are numbered according to Table 1.

Table 3 Performance test results of samples prepared under different process parameters

Serial Number	Mean Thickness /mm	Surface Flatness /mm	Viscous Area Ratio /%	Tensile Strength /MPa	Compare the change amplitude of 1#	Elongation at break/%	Comparison of Change amplitude with 1#
PP	1.005	0.007 1	10	10.68	35%	74.1	33%
7	0.097	0.010	10	7.89	/	55.6	/
11	0.118	0.010	10	7.51	/	23.9	/
9	0.091	0.009	10	6.99	/	27.0	/
2	0.082	0.005	40	6.28	/	10.1	/

Figure 5 shows the surface micromorphology of the low-viscosity semi-cured adhesive sheet produced with optimized process parameters. Scanning electron microscopy reveals that the surface micromorphology of the adhesive sheet is significantly reduced under 1000x and 200x magnifications, with no microcracks observed. Compared to the surface micromorphology of the samples with the four process parameters in Figure 1, there is an improvement effect.

4 Conclusion

(1) The scanning electron microscopy results indicate that after matching adjustments were made to tension, vehicle speed, and adhesive viscosity (with a parameter combination of 25 N, 13 m/min, and 410 cps), the microcracks on the surface of the bonded sheet were significantly reduced when

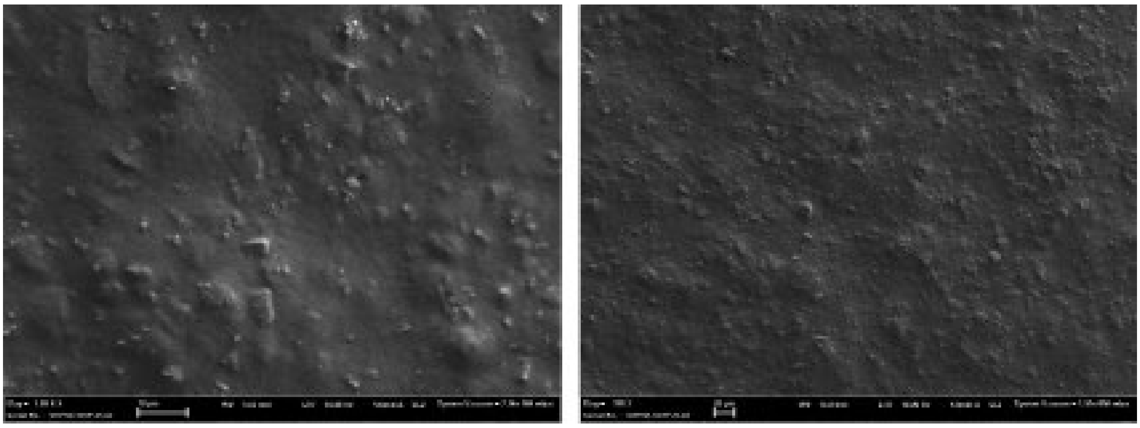

Figure 5 Surface micro-morphology of the sample

observed at a magnification of 1,000 times. When observed at a magnification of 200 times, the microstructure appeared continuous and compact, with good surface quality.

(2) Analysis of DOE experiments and confirmatory experimental data indicates that tension has a significant impact on the mean thickness, but a slightly smaller impact on thickness uniformity; both vehicle speed and adhesive viscosity have a certain impact on both mean thickness and thickness uniformity; tension and vehicle speed also affect the viscosity of the material, and there is a certain interaction between tension and vehicle speed. When making adjustments, it is important to focus on the matching of process parameters such as tension, vehicle speed, and adhesive viscosity.

(3) Based on the influence of main effects and interactions of various factors, the DOE response optimizer was employed to identify a more optimal parameter combination. Under the process parameter combination of a tension of 25 N, a speed of 13 m/min, and a glue viscosity of 410 cps, the produced adhesive sheet material exhibited a thinner thickness level (mean thickness of 1.005 mm), good surface flatness (standard deviation of thickness of 0.0071), and an extremely low viscous area ratio (10%). Notably, the tensile strength reached 10.68 MPa, representing an increase of over 35%, and the elongation at break reached 74.1%, representing an increase of over 33%.

(4) The research results have achieved the joint optimization of multiple indicators such as surface quality, viscosity, and mechanical properties, providing a feasible process plan for the industrial production of low-viscosity and high-flatness adhesive sheets.