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Effect of Curing Agents on Electrical Properties of Low-Temperature Curing Conductive Coatings and Thermodynamic Analysis

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Abstract: Silver-based high-conductivity coatings are used in many advanced manufacturing equipment and components, and existing coatings require high-temperature curing. This paper studies the effects of different curing agents on the electrical properties of low-temperature curing (<100 °C) conductive coatings, and analyzes the effects of different curing temperatures and curing time on the surface resistance, square resistance and resistivity of conductive coatings. The response surface method in Design Expert was used to construct the model, and the curing thermodynamics of different curing agents were analyzed by DSC. It was found that curing agents with lower Tm and activation energy, higher pre-exponential factor and more flexible segments are beneficial to the preparation of highly conductive coatings.

Keywords: curing agent; conductive coating; silver; curing thermodynamics; low-temperature curing

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1. Introduction

Conductive paint is a kind of paint that is applied to the non-conductive bottom plate to make it conductive. Conductive coatings can be divided into two types: intrinsically conductive coatings and filled conductive coatings. In filled conductive coatings, silver has the characteristics of high conductivity (even silver oxide has certain conductivity), and in some industries (aircraft, micro-electronics, electronic printing, etc.) is widely used [1].

The composition of the filled conductive coating can be divided into matrix resin, conductive filler, curing agent, solvent and auxiliary agent. The current research on silver conductive coatings mainly focuses on conductive fillers—namely, silver powder. The content of silver powder [2], the form of silver powder (flower-shaped [3], flake, spherical, linear, etc. [4–6]), the particle size of silver powder (nano-silver ball, nano-silver linear, submicron silver, different sizes of micron silver powder, etc. [4,6–11]) have a great influence on its conductivity. Many researchers have also discussed the influence of different proportions of several silver fillers on the electrical properties of conductive adhesives, such as the blending of spherical silver powder with flake silver powder [4,6–10], and the blending of nano-silver powder with micro-silver powder [12–16]. They also studied the influence of different substances in modified silver powder on electrical properties, such as the effect of modifying the surface of silver powder with DTT, organic acids, long-chain fatty acids, silane-coupling agents and other substances [2,15,17–24]. The influence of matrix resin has also been studied. Studies have found that usually the film-forming resin is polyurethane, which has better electrical properties than epoxy resin [25–27]. The curing process [28] has also been studied.

The curing agent currently studied is mainly high-temperature curing, and the curing temperature is above 120 °C. Wang [29] studied three different types of curing agents—

dicyandiamide, p-hydroxyphenylacetic acid and tetraethylenepentamine—to prepare conductive adhesives. The study found that the curing agent and the substrate have a great influence on the low-temperature curing silver-conductive adhesive. On the glass substrate, the sample has the lowest resistivity of nearly 0.4 mΩ·cm in the experiment (cured at 120 °C). Generally, high-temperature treatment is an important way to obtain excellent electrical properties. Wan [16] obtained a silver coating with a square resistance of 2.1 mΩ/□ at 850 °C, Zhan [30] used a silver wire to prepare a coating with a resistivity of 0.108 mΩ·cm (cured at 140 °C) and Li [31] used silver powder to prepare a coating with a sheet resistance of 8.2 mΩ/□(cured at 150 °C). Yang [32] used molecular simulation methods to establish two molecular reaction mechanism models of amine groups and epoxy groups at the beginning of the curing reaction and compared them with experimental data. He found that, in the initial stage of the curing reaction, the energy barrier of the reaction between the amino group and the epoxy group under the hydroxy-catalyzed condition was reduced. ASome studies have found that even if the sample has been completely transformed during the initial curing process, when the temperature is close to the glass transition temperature (Tg), a post-annealing effect [33] may occur that can further reduce the resistivity.

Among the four main components of coatings (filler, resin, solvent and curing agent), the curing agent has an important effect on performance, especially for epoxy resin systems. For some practical application scenarios, high-temperature curing conditions cannot be provided, but higher electrical properties are required. In order to explore the effect of the curing agent and curing process when curing at low temperature (<100 °C) in this study, four low-temperature (<100 °C) curing agents were used to study the effects of curing temperature and curing time on the electrical properties of the prepared conductive coatings and analyze their curing thermodynamics.

2. Experimental

2.1. Materials and Devices

In this research, we used polyurethane-modified epoxy resin as the matrix resin, and used dibasic acid-treated micron-sized silver tungsten powder (laboratory self-made, the mass ratio of silver to tungsten is 10:1) as the conductive filler (65 wt%). We added a certain amount of diluent, fumed silica and orientation agent, and then a uniformly dispersed conductive puree (component A) was prepared by a high-speed mixer at 1500 r/min for 2 h. Four kinds of the curing agents to cure component A, MH-112 (modified aliphatic amine curing agent), EK315 (a polyamide curing agent), 2060 (cardanol-modified amine curing agent) and TU-2 (long-chain fatty acid amido amine curing agent, laboratory self-made) were used, respectively.

The main test instruments in the experiment were a ST2263 dual-electricity digital four-probe tester (Suzhou Jingge Electronics Co., Ltd., Suzhou, China), HS-DSC-101B differential scanning calorimeter (Shanghai Hesheng Instrument Technology Co., Ltd., Shanghai, China) and an FTIR Affinity-1S infrared spectrometer (Shimadzu, Kyoto, Japan).

2.2. Preparation of the Samples

After mixing the component A and each curing agent according to the r = 1 configuration, they were sprayed on a 50 mm × 100 mm glass test plate with a spray gun. The sample plate was pre-reacted at room temperature for 2 h, and then placed in an oven at 40 °C, 60 °C and 80 °C, and heated for 2 h, 4 h, 6 h and 8 h, respectively, and then the test plate was taken out for testing. The thickness of the prepared paint film after drying was 50 ± 5 μm.

2.3. Test Experiment

The surface resistance, square resistance and surface resistivity of the paint film were tested by the four-probe method. We used DSC to test the Tm value of each curing agent and resin, to calculate the apparent activation energy of the curing process and to analyze the curing thermodynamics of different curing agents.

3. Results and Discussion

3.1. DSC Test and Curing Thermodynamic Analysis

As shown in Figure 1, it can be seen from the DSC curve that the curing temperature Tm of the modified fatty amine curing agent MH112 and cardanol-modified amine curing agent 2060 was lower, followed by fatty acid amido amine curing agent TU-2 and polyamide curing agent EK315. The Tm value is shown in Table 1. Overall, 2060 emitted the highest heat during the curing process.

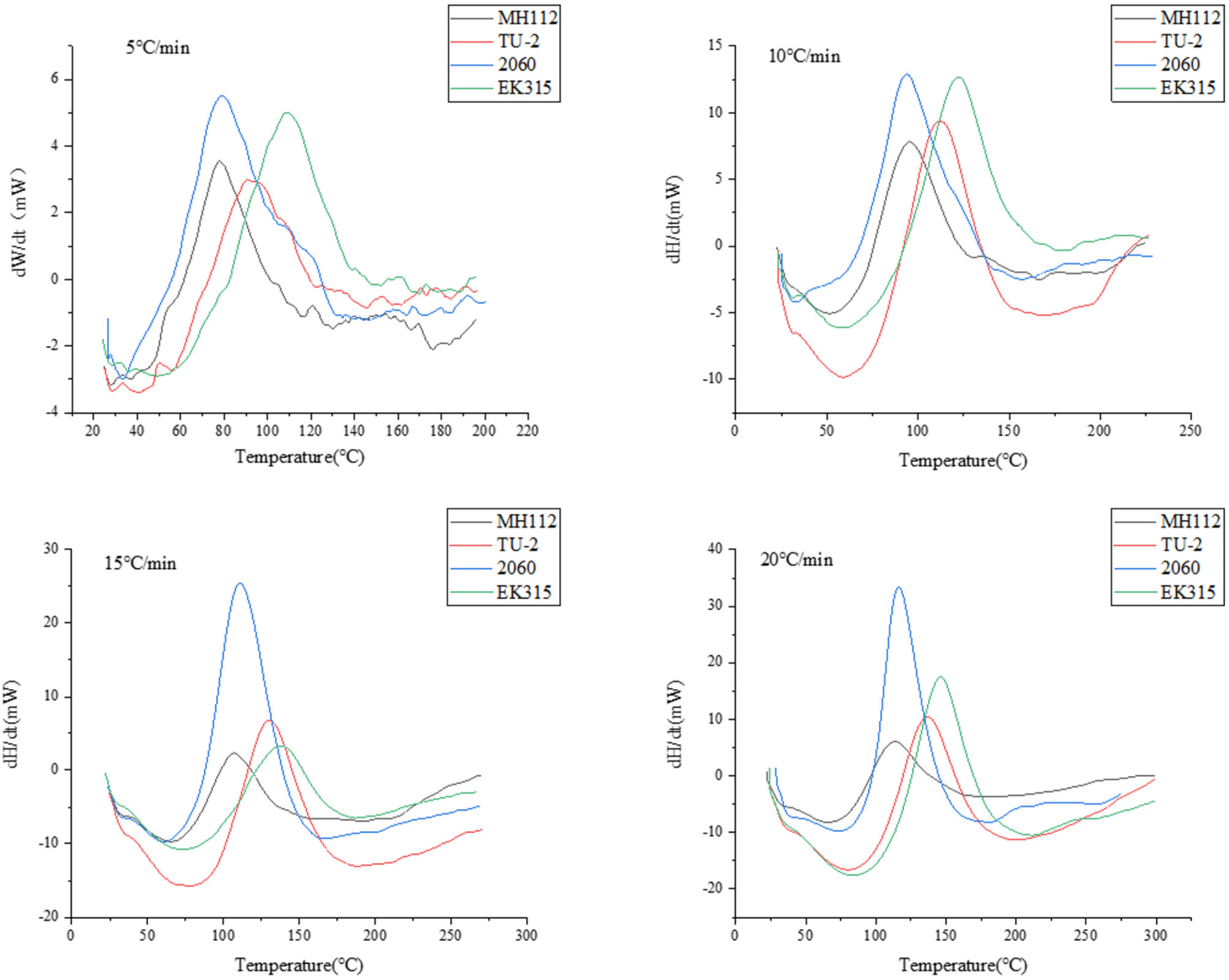

Figure 1. DSC curves with scan rates of 5, 10, 15 and 20 °C/min.

Table 1. DSC test peak temperature of different heating rates.

Heating Rate (°C/min)	Peak Temperature (°C)			
	MH112	TU-2	2060	EK315
5	78	90.9	79.2	109.6
10	95.4	112.2	93.9	122.4
15	106.6	129.9	110.7	137.4
20	113.7	136.9	116.4	146.2

According to the data in Table 1, the calculation and analysis of curing thermo-kinetics were carried out. Through the Kissinger equation $\ln\frac{\beta}{T_p^2} = \ln\frac{AR}{E_k} - \frac{E_k}{R}\cdot\frac{1}{T_p}$, plot $\ln\frac{\beta}{T_p^2}$ and $\frac{1}{T_p}$, to perform linear fitting (Figure 2) and calculate the apparent activation energy and pre-exponential factor of each curing agent.

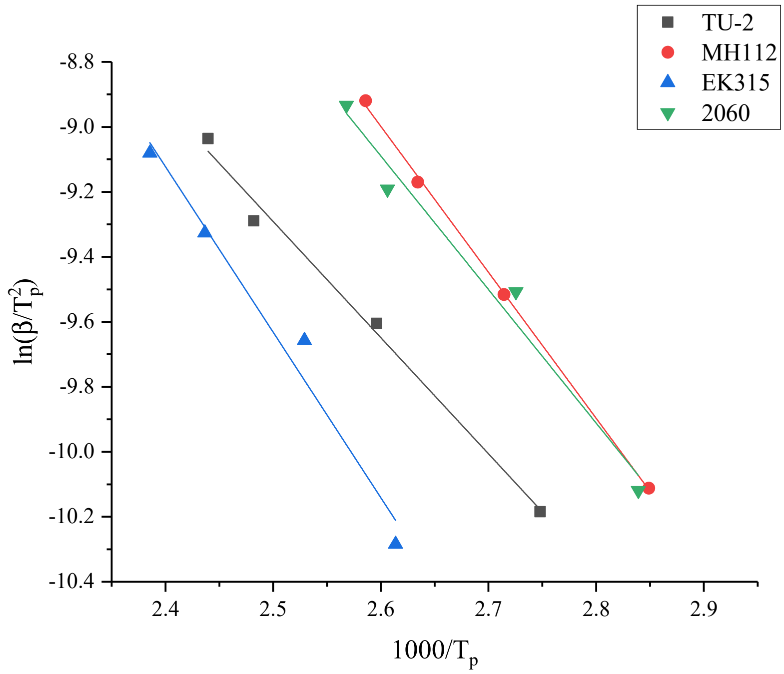

Figure 2. Fitting curve of reaction activation energy and pre-exponential factor of curing with different curing agents.

We used the crane equation $\frac{d(ln\beta)}{d\left(\frac{1}{T_p}\right)} = -\frac{E_k}{nR}$, plot $ln\beta$ and $\frac{1}{T_p}$ (shown in Figure 3) to calculate the curing reaction order. The calculation results are shown in Table 2.

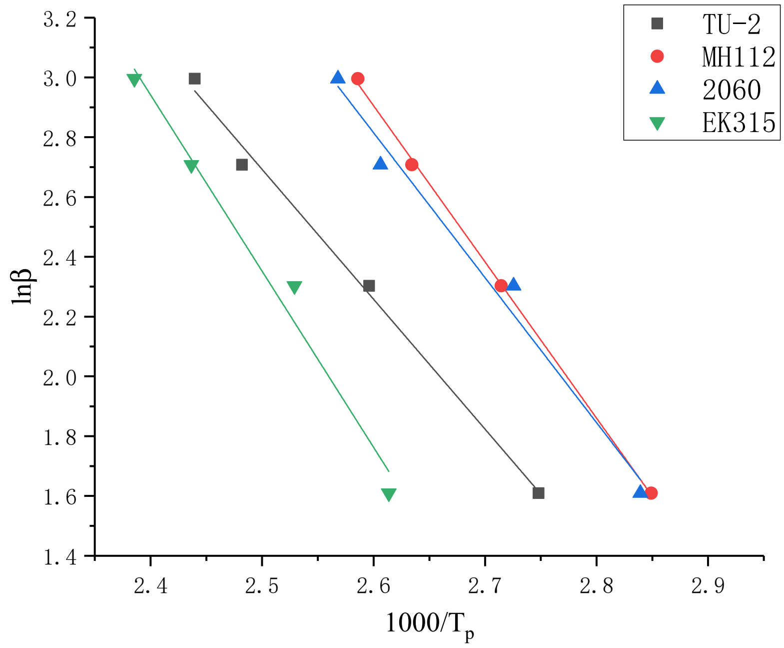

Figure 3. Fitting curve of reaction order of different curing agents.

Table 2. Reaction kinetic parameter values of different curing agents.

Curing Agent	MH112	TU-2	2060	EK315
apparent activation energy (kJ/mol)	37.35	29.72	34.17	42.35
pre-exponential factor	65,641	5094	20,287	113,131
reaction order	0.86	0.82	0.85	0.86

It can be concluded from Table 2 that the reaction orders of the four curing agents are similar, and the essence of the curing reaction is the same. The apparent activation energy of TU-2 was the lowest, but the activation energy of EK315, which is also amide-cured, was the highest, indicating that the introduction of long-chain fatty acids reduces the activation energy. The activation energy of 2060 and MH112 were close, but the pre-exponential factor of MH112 was about 3 times that of 2060.

Both 2060 and MH112 are aliphatic curing agents for primary amines, so the apparent activation energy is low. When the epoxy is cured by reaction, a denser three-dimensional network system can be formed, which makes the contact between conductive fillers close and enhances the conductivity of the coating. TU-2 and EK315 are amide-based curing agents, and TU-2 is obtained by modifying fatty acids. There is a certain amount of fatty acid in TU-2 curing agent as a catalyst during curing, which greatly reduces the activation energy during curing.

The organic acid catalysis mechanism is shown in Figure 4. During the curing reaction, the organic acid acts as a proton donor. The carboxyl group first forms a hydrogen bond with the epoxy group, and then forms a tri-molecular transition state with the amine curing agent. In this state, the epoxy group opens the ring, rapid proton transfer occurs, the O–H on the carboxyl group is broken, the carboxylate ion combines with the hydrogen on the amine and the N–H bond breaks to form an organic acid again.

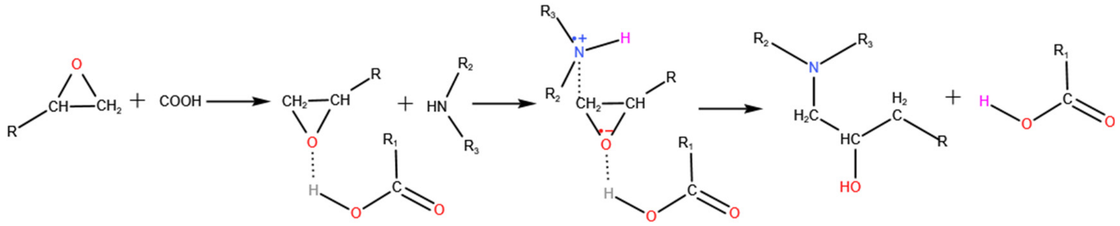

Figure 4. Catalytic reaction mechanism.

3.2. FTIR Text

Figure 5 shows the FITR results of the four curing agents. It can be seen from the figure that each curing agent has many peaks in the spectrum, which means that there are very different functional groups that make it easy for other reagents to react and change the electrical properties of the conductive coating. Tu-2 and EK315 have more similar functional groups, and MH112 and 2060 have more similar functional groups.

Furthermore, the spectrum shows that TU-2 and MH112 have a strong absorption peak near 1100 cm^{-1}, which is a flexible C–O–C chain, which makes the molecular chains of these two curing agents easier to move and arrange during the curing process. As a result, the conductive filler can be better arranged on the surface layer, so as to obtain better conductive performance.

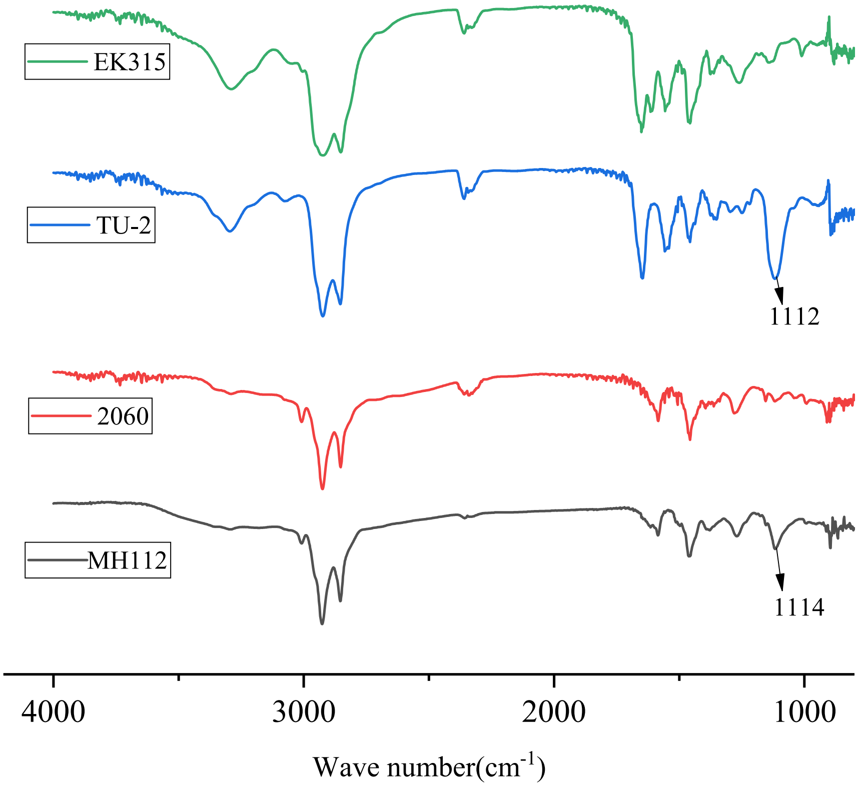

Figure 5. FTIR of four types of curing agents.

3.3. *Surface Resistance*

The samples with curing agents MH-112, EK315 and TU-2 did not sufficiently cure after heating at 40 °C for 2 h, and the instrument did not successfully test the data. Thus, there are no data for these samples. The surface resistance results of the curing time and curing temperature are shown in Figure 6.

It can be concluded from Figure 6 that the surface resistance of these four curing agents is showing a downward trend with the increase of curing temperature and extension of curing time. This is because an increase in temperature is beneficial to increase the curing rate, and extending the curing time increases the degree of reactive curing. However, when MH-112 and 2060 were cured at 80 °C, the curing time extended from 2 h to 8 h. The surface resistance changed very little. From the DSC test and thermodynamic analysis of the two resins at 5 °C/min, it can be seen that the Tm was around 80 °C and the activation energy was low, so the curing was probably sufficient when the reaction is at 2 h.

At the same curing temperature and curing time, the surface resistance of the coating cured by MH112 was the lowest, followed by 2060 and YU-2 curing, and the highest was cured by EK315. This is because the Tm and activation energy of MH112 and 2060 are low and the pre-exponential factor is high, and there may be a post-annealing effect [31] to improve the conductivity. EK315 had the highest pre-exponential factor, but also the highest activation energy and Tm value, and it is thus difficult to start curing at a lower temperature.

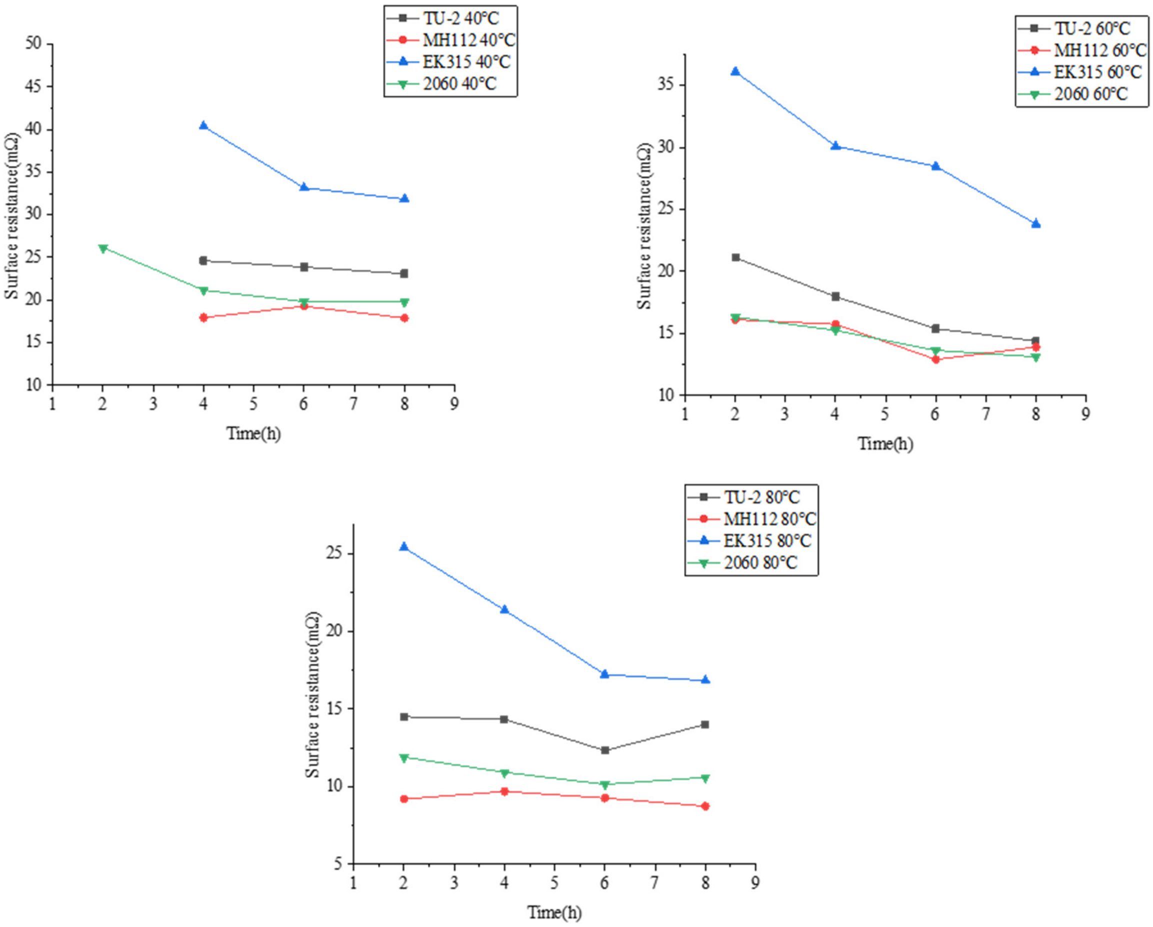

Figure 6. The relationship between the samples' surface resistance(Y axis) and curing time(X axis).

Although the activation energy of MH112 was slightly higher than 2060, the pre-exponential factor of MH112 was higher. Compared with 2060, MH112 has a C–O–C chain and a longer flexible chain. It can move better during curing and shrinkage. The conductive filler can be better arranged on the surface, which improves the surface conductivity. The conductive properties of the flexible amide-based curing agent TU-2 are also better than those of the polyamide curing agent EK315.

For further analysis, we used the response surface method in Design Expert to analyze the data.

As shown in Figure 7, in the studied temperature and time range, the 3D image can be seen as a plane, and the effect of the curing temperature and time on the surface resistance is close to a linear relationship. Therefore, using the recommended 2FI model, the predicted R^2 value was 0.8573, close to the actual R^2 value, and the model is credible (R^2 value is the model correlation coefficient). The relationship between surface resistance, curing time and curing temperature can be obtained by processing, as shown in the table below.

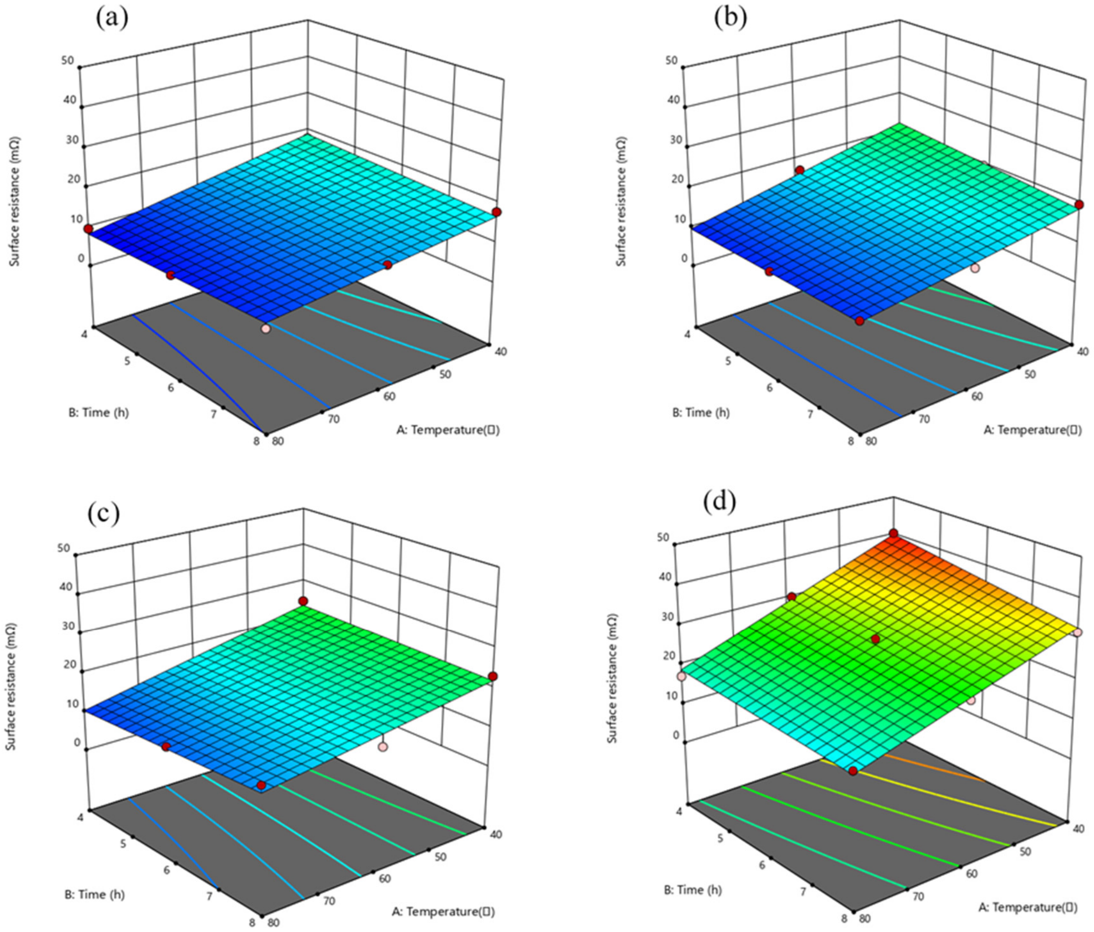

Figure 7. 3D diagram of surface resistance, curing time and temperature ((**a**) MH112; (**b**) 2060; (**c**) Tu-2; (**d**) EK315; A is the curing temperature (°C); B is the curing time (h); Y axis is surface resistance (mΩ)).

The *p*-value of A was 0.0001 and the *p*-value of B was 0.0215. The smaller the *p*-value, the more significant the effect. So, the curing temperature of the curing agent is the most important factor, and the curing time has little effect on the surface resistance. It can be seen from Table 3 that the constant of MH112 of aliphatic amine was the lowest, and the constant of polyamide curing agent EK315 was the highest. The rigid modification group in 2060 causes the constant to increase, while the flexible chain in TU-2 can effectively reduce the constant value. The higher the Tm value of the curing agent, the greater the effect of curing temperature and curing time on the surface resistance, so the surface resistance of EK315 changes the most. The curing time had the least influence on the surface resistance of the flexible MH112, curing at 80 °C for 2 h it was 9.2 mΩ, and after 8 h it reached the minimum value of 8.7 mΩ.

Table 3. Model equation of surface resistance.

Surface Resistance /mΩ	Model Equation
MH112	R = 36.3304 − 0.3735A − 1.6299B
2060	R = 42.5172 − 0.4181A − 1.9434B
TU-2	R = 41.9120 − 0.4289A − 1.3906B
EK315	R = 72.5803 − 0.6339A − 2.8378B

Note: R is the surface resistance (mΩ); A is the curing temperature (°C); B is the curing time (h); *p*-value is the factor significance coefficient.

3.4. Square Resistance

From Figure 8, it can be seen that all curing agents can significantly reduce the square resistance of the prepared conductive coatings by increasing the curing temperature. After MH112 is cured for 2 h, the square resistance can be changed little by prolonging the curing time. When 2060 is cured at 40 °C, the square resistance is reduced by prolonging the curing time, and the square resistance is lower when curing at 60 °C and 80 °C for the longer curing time. TU-2 and EK315 with higher Tm extend the curing time and decrease the square resistance greatly. The lower the curing temperature, the more significant the extension of the curing time for reducing the square resistance. In the curing experiment at 80 °C, the square resistance of MH112 was the lowest at 41 mΩ/□, and it had excellent electrical conductivity.

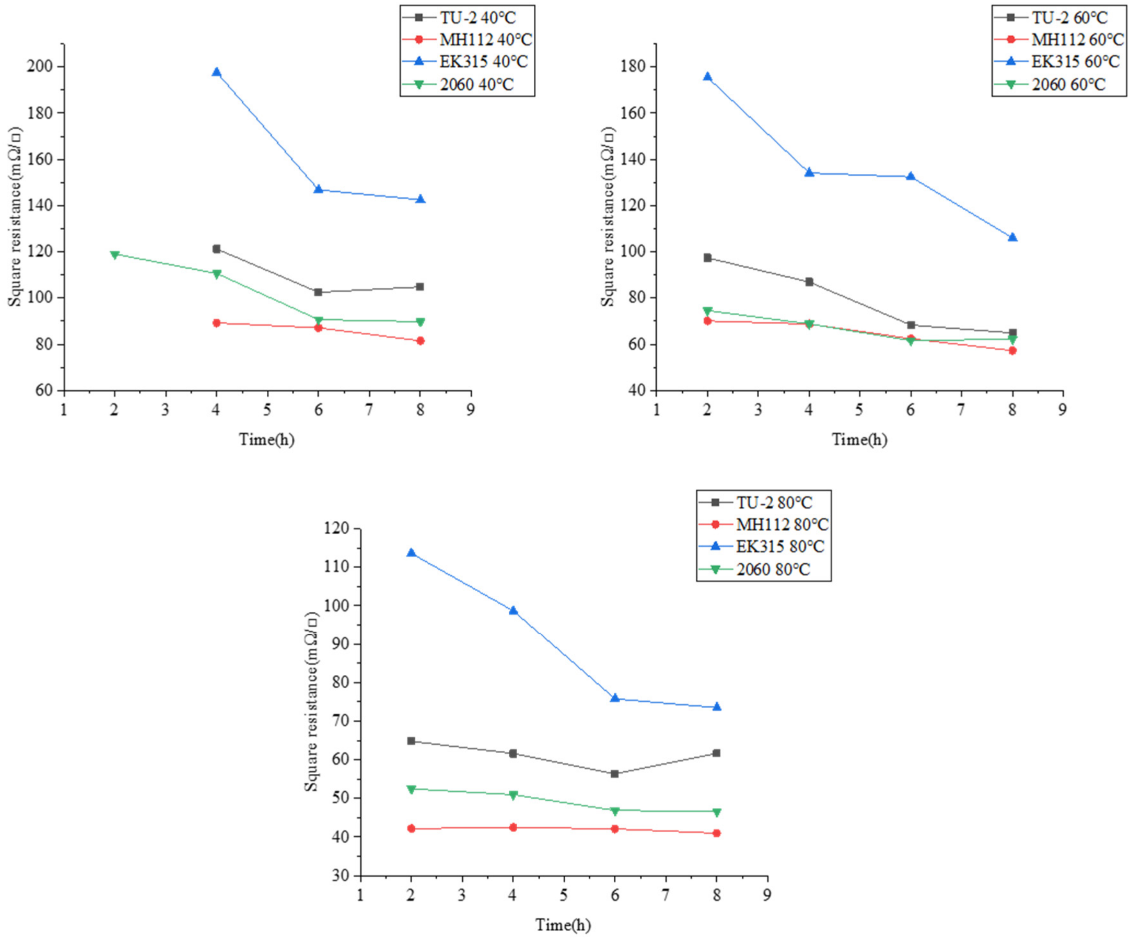

Figure 8. Relation between square resistance (Y axis) and curing time (X axis).

Using the response surface method in Design Expert to analyze the data, the relationship equations between sheet resistance, curing temperature and curing time were identified and are shown in Table 4.

Table 4. Model equation of square resistance.

Square Resistance (mΩ/□)	Model Equation
MH112	$R = 199.2142 - 2.1775A - 11.9581B$
2060	$R = 219.7686 - 2.3685A - 12.4361B$
TU-2	$R = 230.9301 - 2.5396A - 11.0300B$
EK315	$R = 374.9305 - 3.4845A - 18.1160B$

Note: R is the surface resistance (mΩ/□); A is the curing temperature (°C); B is the curing time (h); *p*-value is the factor significance coefficient

The *p*-value of A was 0.0001 and the *p*-value of B was 0.0058. It can be obtained from the model equation where the constant of the amide-based curing agent is higher than that of the fatty amine, because the activity of the secondary amine is lower than that of the primary amine. The model equation constant value of 2060 was higher than that of MH112 because the aromatic structure increases the sheet resistance after curing. The constant of TU-2 was significantly lower than that of EK315. This is because the Tm value is lower and the flexible structure is more flexible and the apparent activation energy is lower.

3.5. Resistivity

The measured resistivity of samples prepared with different curing agents at different curing temperatures and curing times is shown in Figure 9, and the model equations obtained by the response surface method are shown in Table 5.

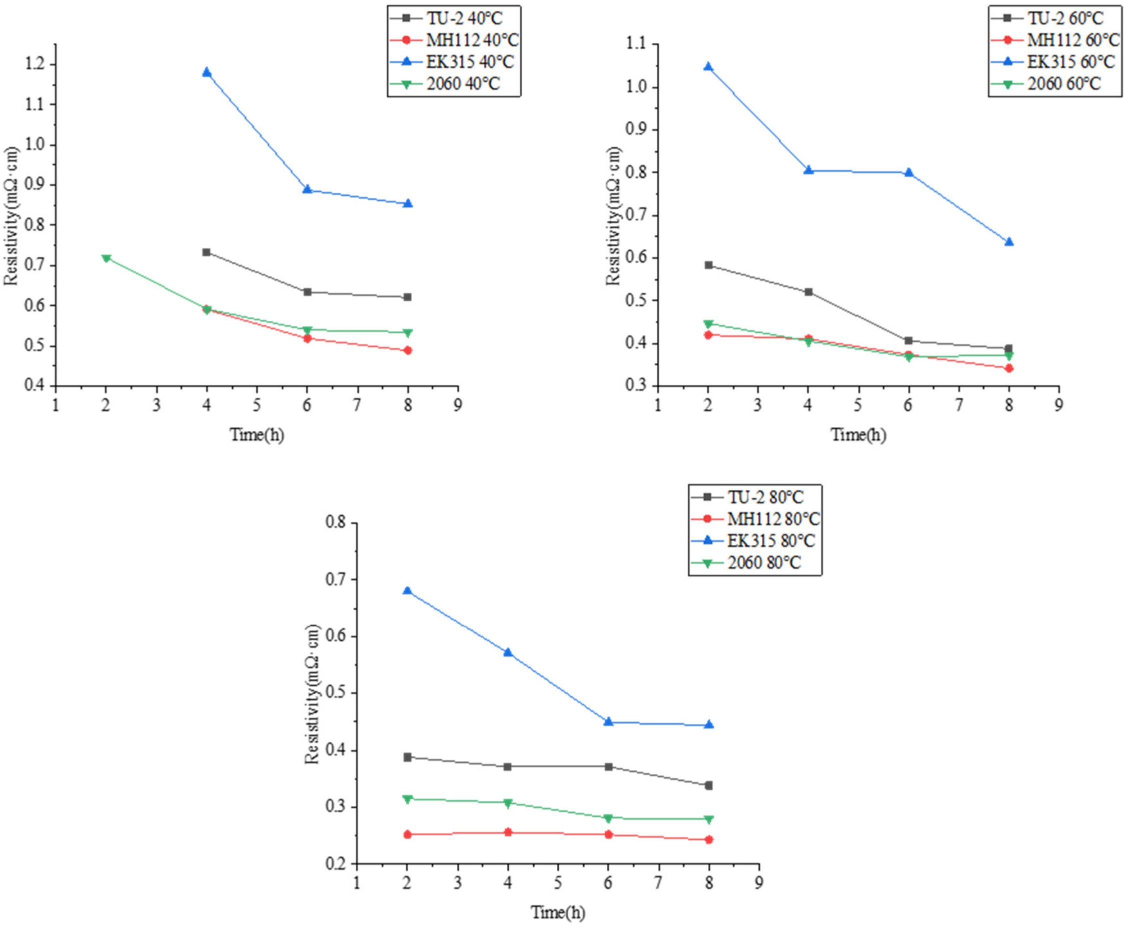

Figure 9. Resistivity of the samples (X: curing time; Y: Resistivity).

Table 5. Model equation of resistivity.

Resistivity (mΩ·cm)	Model Equation
MH112	$R = 1.3395 - 0.01457A - 0.08688B$
2060	$R = 1.3644 - 0.01506A - 0.08217B$
TU-2	$R = 1.4392 - 0.01627A - 0.07288B$
EK315	$R = 2.2860 - 0.02164A - 0.1156B$

Note: R is the surface resistance (mΩ·cm); A is the curing temperature (°C); B is the curing time (h); *p*-value is the factor significance coefficient.

The *p*-value of A was 0.0001 and the *p*-value of B was 0.004. For a kind of curing agent, it can be seen from the figure that the effects of curing temperature and curing time on resistivity are the same as their effects on surface resistance and square resistance. The curing temperature is the main influencing factor. Increasing the curing temperature can significantly reduce the resistivity, and extending the curing time reduces the resistivity relatively little. Some researchers [31] believe that the curing temperature dependence of resistivity is caused by the difference in internal stress generated during the curing process. Because the higher temperature is most conducive to the curing reaction, the curing rate is increased, and a dense conductive coating is obtained. It can be seen from Table 5 that the curing agent constant term of the primary amine was lower than that of the secondary amine curing agent; because the activity of the primary amine is higher, the curing agent constant of the flexible chain will also be reduced (MH112 was lower than 2060, TU-2 was lower than EK315). The structure of the curing agent affects the activation energy and pre-exponential factor of curing. Conductive coatings prepared with lower activation energy and a higher pre-exponential factor have lower resistivity. The influence of several times the pre-exponential factor gap is similar to the lesser activation energy difference. In the experiment, the lowest resistivity of MH112 after curing was 0.24 mΩ·cm, the lowest of 2060 curing was 0.28 mΩ·cm, the lowest curing of TU-2 was 0.33 mΩ·cm and the lowest curing of EK315 was 0.44 mΩ·cm. Their electrical properties were better than the conductive adhesives obtained by curing epoxy with triethanolamine [8] at 200 °C (0.789 mΩ·cm, 67 wt% silver flakes).

4. Conclusions

This study mainly discusses the effect of curing agent on low-temperature curing silver conductive coatings. The experiment passes the same conductive paste to react with four curing agents—MH112, 2060, TU-2 and EK315.—after curing for 2 h, 4 h, 6 h and 8 h at 40 °C, 60 °C and 80 °C, respectively. We measured the surface resistance, square resistance and resistivity of the conductive coating to study the effect of curing temperature and curing time on the conductivity of the coating under curing at <100 °C. We used the DSC test to calculate the apparent activation energy, pre-exponential factor and reaction order of each curing agent, and analyzed its curing thermodynamics.

The experiments in this study found that the influences of investigating factors on surface resistance, sheet resistance and resistivity are consistent. Experimentally found, the curing temperature is the most significant influencing factor. Increasing the curing temperature will significantly reduce the electrical properties, such as surface resistance, square resistance and resistivity, of the conductive coating. When the curing temperature is much lower than the Tm value, extending the curing time can greatly improve the electrical properties of the conductive coating. However, if the curing temperature is close to the Tm value, extending the curing time will not significantly improve the electrical properties of the coating. For example, the surface resistance of MH112 after curing for 2 h was about 9.2 mΩ, but the surface resistance after curing for 8 h was also 8.7 mΩ.

The structure of the curing agent affects the electrical performance of the coating. The electrical performance of the coating prepared by the primary amine curing agent is better than that of the secondary amine curing agent, and the flexible chain can improve the electrical performance of the conductive coating.

Through the thermodynamic analysis of the curing agent, the pre-exponential factor and apparent activation energy affect the electrical properties of the cured conductive coating. Among them, apparent activation energy is the most important influencing factor, and the pre-exponential factor has less influence. Conductive coatings prepared by curing agents with lower activation energy have better electrical properties. The pre-exponential factor has little effect on the electrical properties of the conductive coating, and the pre-finger factor that is several times different will significantly change the electrical properties of the conductive coating.

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