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Synthesis and characterization of elastomeric polyester coatings for automotive pre-coated metal

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ABSTRACT

An automotive pre-coated metal system (PCM) has been investigated to replace wet coating process, such as pre-treatment, dip coating and spray coating, to address environmental regulations. However, automotive pre-coated metal sheets must have high flexibility and stiffness to overcome harsh conditions such as cutting, press and stamping processes. For these reasons, elastomeric polyester coatings were designed to improve scratch resistance and to impart reflow characteristic for an automotive PCM. The characteristics, curing behavior and viscoelastic property of the resins were measured by ^{1}H NMR, GPC, RPT and DMA. The flexibility was evaluated using a texture analyzer. A nano scratch tester, equipped with an optical microscope was used to measure the scratch resistance of coatings and the scratched surface morphology. An Amtec laboratory car wash test and a sharp knife were used to evaluate the reflow property of the coatings. Change of the damaged surface morphology was measured using a mini-SEM.

With increasing 1,4-cyclohexanediol content, the final frequency decreased and the storage modulus and T_g of the coatings increased. This is related to the high stiffness of the cyclohexane structure of 1,4-cyclohexanediol. In terms of scratch resistance, EP-6, one of the formulations examined, has high scratch resistance due to the restoring force of the stiff structure of 1,4-cyclohexanediol. Consequently, the gloss recovery after a heat treatment was increased with increasing the content of cyclohexane structure in the car wash test. In the case of a deep scratch, the reflow property of coating was demonstrated using a heat treatment. Therefore, a cyclohexane structure is a good factor for improving the scratch resistance and reflow property of the polyester coatings.

1. Introduction

Scratch damage is an essential type of paint damage from environmental causes, and is a major concern and warranty problem in the automotive industry. In the case of car washing, dusts embedded in brush materials can cause numerous micro-scale scratches on the coating surface. The overall effect is sometimes referred to as swirl marks. These micro-scale scratches cause an early change in appearance and loss of gloss on the coating surface. The cost of automotive coating warranty related to environmental causes has been estimated to be $2 billion a year worldwide [1,2]. Therefore, extensive studies have been carried out to solve the problem, and the methods used can be categorized into two approaches. The first is to control the surface hardness of coatings for good scratch resistance. However, hard surfaces easily crack and are prone to damages by thermal shock and adjacent scratching. In comparison, soft surfaces have lower mar resistance and gloss. The second approach to increasing scratch resistance is to control the reflow property of coatings. As shown in Fig. 1, stiff segments and flexible chains of the coating are extended in the same layer. This structure can restore scratches from an environmental deformation [3].

Pre-coated metal system (PCM) is manufactured in a sheet or coil coating line and processed and assembled in factories for household electric appliances, building materials and others. In this system, wet coating processes can be eliminated by using a roll coating process, thereby the problem of solvent evaporation can be solved. In addition, a pre-coated metal system offers other advantages such as improved productivity and energy saving. Due to these reasons, automotive pre-coated metal systems have been investigated to remove the wet coating process, such as pre-treatment, dip coating and spray coating. In this system, all coating layers must have high flexibility and formability to overcome harsh conditions due to the cutting, pressing and stamping processes [4–9].

In one of our previous studies, polyester resins were synthesized with polycarbonatediol to control the flexibility for an

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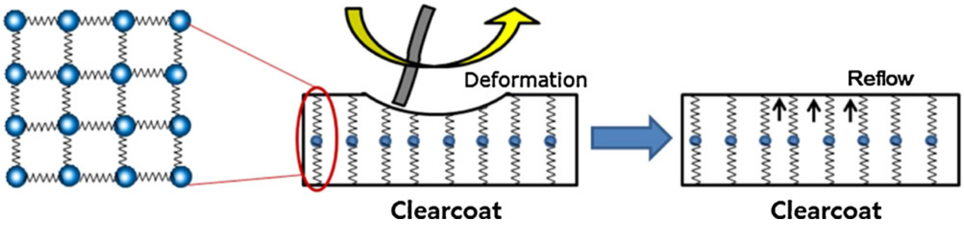


Fig. 1. Schematic view of the reflow mechanism for automotive clearcoat [8].

automotive PCM. The resins were designed to demonstrate the flexible chain effect, using polycarbonatediol. Results showed that the synthesized polyester coatings had high elongation values and good scratch resistance for the stamping, pressing and cutting processes in an automotive PCM [9]. In this study, we investigated an elastomeric polyester resin system to improve the scratch resistance and the reflow property of an automotive PCM. The resins were designed to increase the stiffness of coatings using 1,4-cyclohexanediol. Cyclohexane is a stiff molecule of chair form consisting of C—C single bonds which have a maximum rotational barrier at ±60°. For this reason, cycloheptane and cyclooctane are soft molecules in comparison with cyclohexane [11,12]. The elastomeric polyester resins were synthesized with an optimized polycarbonatediol content and different 1,4-cylcohexanediol contents. The curing and viscoelastic behavior, flexibility, scratch resistance and reflow property were measured to determine the effect of a cyclic structure for an automotive PCM.

2. Experimental

2.1. Materials

1,4-Cyclohexanediol (Tokyo Kasei) was used to improve the stiffness of coatings. Polycarbonatediol (PCDL, $M_w = 1000$, Asahi Kasei Co.), trimethylol propane (TMP, Tokyo Kasei), isophthalic acid (IPA, Junsei Chemical), phthalic anhydride (Daejung Chemical), ethylene glycol (EG, Samchun Chemical) and 1,6-hexandiol (Samchun Chemical) were used without further purification.

Hexamethoxymethylmelamine (HMMM, Cytec), a curing agent in pre-coated metal systems, was used. An amine type curing catalyst (NACURE 5925) was supplied from KING industries. Butylstannoic acid (FASCAT 4100, Arkema Inc.) was used as a catalyst to catalyze polymerization and prevent a transesterification reaction during the synthesis reaction [9]. Table 1 lists the synthesis formulation of the polyester resin. A conventional automotive clearcoat which is an acrylic coating was supplied from PPG Korea, comparing various properties.

2.2. Synthesis of elastomeric polyester resin

2.2.1. Synthesis of polyester pre-polymer

Polyester pre-polymer was synthesized using 1,4-cyclohexanediol, trimethylol propane, 1,6-hexanediol, ethylene glycol, phthalic anhydride and isophthalic acid according to the following process. The resin was designed as a carboxy-excess pre-polymer. A 500 ml 4-neck round type reactor, fitted with a temperature controller, heating mantle, N_2 purge and condenser (for removing the water from esterification) was placed. All raw materials were charged into the reactor and the temperature was set to 160 °C for 1 h. Subsequently, the reaction temperature was increased from 160 °C to 220 °C at 0.5 °C/min. During the synthesis process, the acid value titration was performed using a 0.1 N KOH solution. The carboxy-excess pre-polymer was reacted to an acid value of 20 [9].

Table 1
Formulation of the elastomeric polyester resins (unit: mole of monomer).

Contents	EP-0	EP-2	EP-4	EP-6
Polycarbonatediol	3	3	3	3
1,4-Cyclohexanediol	0	2	4	6
Trimethylol propane	7	7	7	7
Ethylene glycol	3	3	3	3
1,6-Hexanediol	12	10	8	6
Phthalic anhydride	11	10	10	10
Isophthalic acid	12	12	12	12

2.2.2. Synthesis of polyester resin with polycarbonatediol

Starting with the carboxy-excess pre-polymer in the reactor, the temperature was set to 80 °C and PCDL was charged into the reactor with an impeller. Subsequently, the temperature was increased from 80 °C to 180 °C at 0.5 °C/min. During the synthesis process, the acid value titration was performed using a 0.1 N KOH solution. The temperature was maintained and the solvent was added over a period of several hours to remove condensed water and obtain a hydroxy-terminated polyester resin. Scheme 1 shows the synthesis process of the polyester resin [9].

2.2.3. Preparation of polyester coatings for automotive PCM

The polyester coatings were prepared by blending polyester resin and HMMM with catalyst, according to 8:2 ratio. The blended mixture was coated onto a galvanized steel sheet using a 40 μm bar applicator and cured at 150 °C for 30 min. The cured films, prepared for DMA and tensile tests, were coated on a disposable aluminum dish and cured under the same condition. The width and thickness of cured films were 6.5 mm and 0.5 mm, respectively [9].

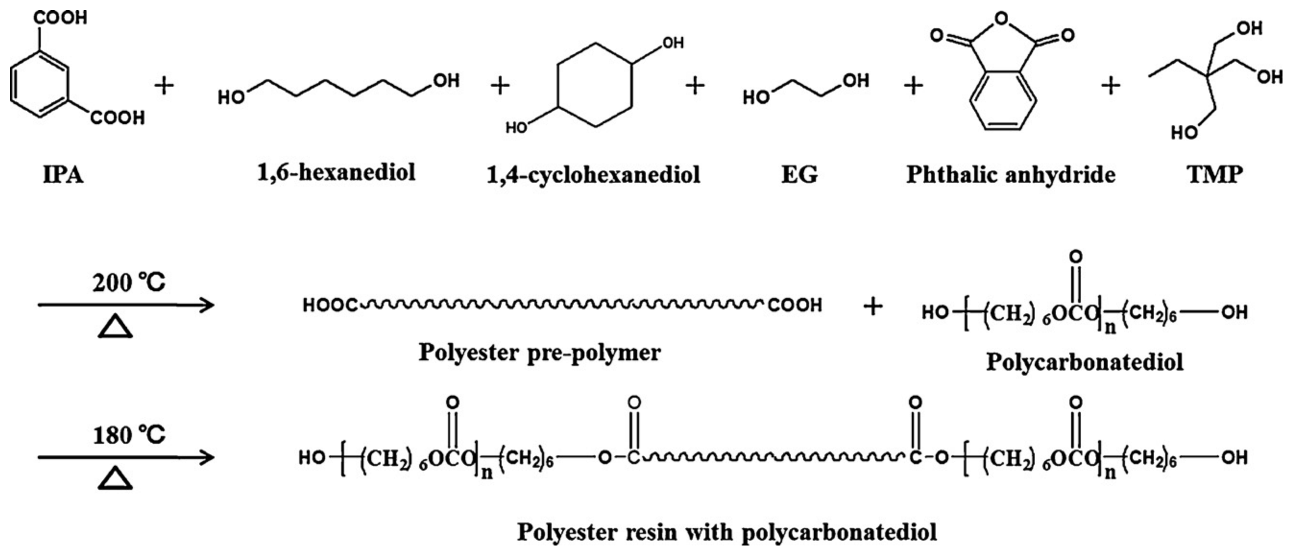
2.3. Methods

2.3.1. Characterization of the elastomeric polyester resin

To characterize the polyester resins ^{1}H NMR spectra were obtained using a 400 MHz NMR spectrometer (Jeol JNM-LA400, JEOL Ltd., Japan). The molecular weight and polydispersity were measured using an YL9100 GPC SYSTEM (Young Lin, Korea) equipped with a pump, a RI detector and a Waters Styragel HR 5E column. Polystyrene and poly(methyl methacrylate) calibration standards were used. Tetrahydrofuran was used as the eluent, and the flow rate was 1 ml/min. Table 2 lists the molecular weight and polydispersity of the synthesized resins [13].

2.3.2. Rigid-body pendulum physical property testing (RPT)

The curing behavior of the polyester resin was measured using a rigid-body pendulum physical property testing instrument (RPT, A&D Co., Japan). Fig. 2 shows two types of rigid-body pendulum – a knife edge type pendulum which is used to measure the curing behavior and a cylindrical edge type to analyze physical properties, such as T_g of the cured coatings. The curing behavior was measured by detecting the movement of a knife type pendulum on the coated steel substrate as a function of temperature. The oscillation pattern of the pendulum is related to the change in surface properties such as chemical or physical networking. This apparatus consists



Scheme 1. Synthesis process of the elastomeric polyester resin.

Table 2
Characterization of the elastomeric polyester resins.

Property	EP-0[b]	EP-2	EP-4	EP-6
Number average molecular weight (g/mol)	7350	6960	7520	7310
Polydispersity (M_w/M_n)	3.93	3.34	2.98	2.86
n_{OH} (mg KOH/g)[a]	70	71	71	71

[a] n_{OH} – theoretical hydroxyl number of polyester resins.
[b] EP-0: data is derived from Ref. [9].

of heating oven, steel substrate and main controller. To examine curing the polyester coatings were coated on the steel substrate (50 mm × 20 mm × 0.3 mm) using a 40 μm coating tool. The heating condition was set at 50 °C for 3 min to stabilize the uncured coatings. Afterward, the temperature was increased from 50 °C to 200 °C at 5 °C/min, and maintained at 200 °C for 50 min [14].

2.3.3. Dynamic mechanical analysis (DMA)

The viscoelastic behavior of polyester coatings is an important factor for evaluating coating performance as a function of temperature. Dynamic mechanical analysis (DMA) was performed using a Dynamic Mechanical Analyzer Q-800 (TA Instruments, USA). The machine was used to determine storage moduli and glass transition temperature. The test method was a tension-film mode under the following conditions: a frequency of 1 Hz, strain of 0.3%, temperature ramp from −60 °C to 160 °C at a heating rate of 2 °C/min [9].

2.3.4. Surface hardness test

To evaluate the change in the surface hardness of each cured coatings, the pendulum hardness of the coatings was measured using a pendulum hardness tester (Ref. 707PK, Sheen Instruments Ltd.) according to the König method (ANS/ISO 1522) at 23 ± 1 °C and 50 ± 2%. The scratch resistance was measured using a No. 553

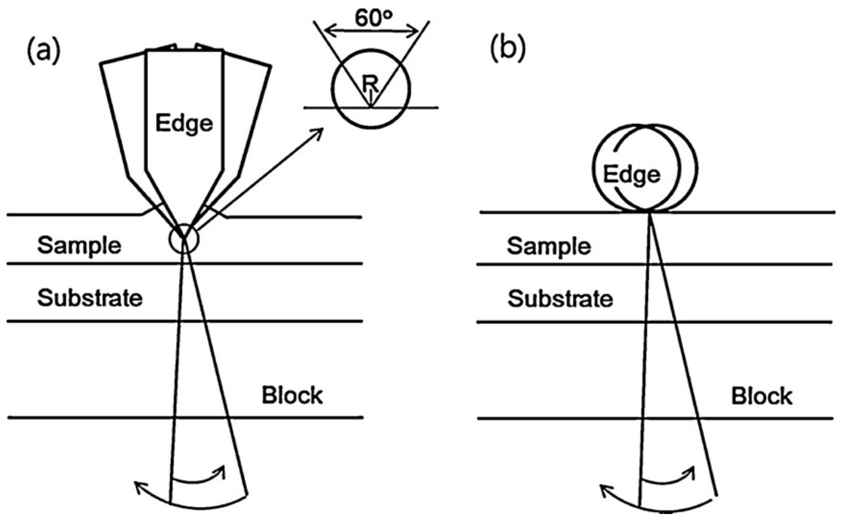


Fig. 2. Rigid-body type pendulums in a RPT instrument: (a) knife edge type for the curing process; (b) cylindrical edge type for physical properties.

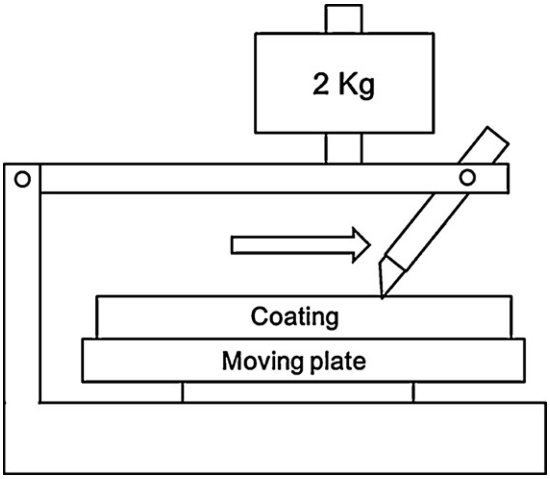


Fig. 3. The concept of a scratch maker to measure reflow property of coatings using an automatic pencil hardness tester equipped with a sharp knife and a 2 kg weight.

pencil hardness tester (Yasuda Seiki Seisakusho Ltd.). The test is in accordance with ASTM D 3363-74 [10].

2.3.5. *Tensile strength*

The flexibility of the polyester coatings was determined from tensile tests using a texture analyzer (TA-XT2i) with rectangle-shaped specimens according to the ASTM D-638-10 method. The tensile strength was calculated by dividing the maximum load in Newtons (N) by the average original cross-sectional area (mm^2) in the gage length segment of the specimen. The percent elongation (strain %) was calculated by dividing the change in gage length by the original specimen gage length, expressed as a percentage (%) [9,15].

2.3.6. *Nano scratch test (NST)*

Scratch resistance is an important property in automotive clearcoat, and is related to gloss retention and weatherability. A nano scratch tester (CSM Instruments, Switzerland) is used to evaluate the scratch resistance or mar resistance of coatings using a diamond stylus scratch method. The machine has several loading modes, the progressive normal force mode and constant normal force mode, which are used to measure the fracture point and penetration depth profile of the coatings. This method is performed according to ASTM D 7187-05, and the progressive normal force mode is carried out using a sphero-conical 90 type indenter. The progressive load applied from 0.2 to 40 mN at a loading rate of 39.8 mN/min [9,16].

2.3.7. *Amtec laboratory car wash test*

Gloss retention is an important factor to maintain the gloss and color of coatings. An Amtec laboratory car wash test with rotating brushes is simulated using quartz powder defined as synthetic dirt. During the test, rotating brushes are moved back and forth at 10 times with the washing solution, consisting of water and quartz powder. After cleaning and drying, the measurement of gloss at 20° geometry was carried out to evaluate the gloss change of coatings. Subsequently, the scratched coatings were placed in a heating oven at 100 °C, for 2 min and a 20° gloss measurement was performed again to evaluate reflow property [5].

2.3.8. *Reflow property*

An automatic pencil hardness tester was used to make a deep scratch on the coating surface. The tester is equipped with a sharp knife and a 2-kg weight as shown in Fig. 3. The damaged coatings were placed in a heating oven at 100 °C for 0, 10 and 30 min. The morphology change of the damaged surface was measured using a mini-SEM (SNE-3000M, SEC Co., South Korea).

3. Results and discussion

3.1. *Characterization of the elastomeric polyester resin*

The synthesized polyester coatings focused on the amount of stiff structure with 1,4-cyclohexanediol. Generally, a commercial automotive clearcoat requires physical properties such as scratch resistance, stone chip resistance and gloss. However, additional properties such as flexibility and stiffness are required to overcome harsh conditions during the cutting, pressing and stamping processes in an automotive PCM [9]. Table 2 lists the molecular weight and polydispersity of the polyester resin as determined by GPC. In our designed formulation, the theoretical hydroxyl number of polyester resins, n_{OH}, was fixed at around 70 to remove the effect of curing agent. The ^1H NMR spectra of EP-0 and EP-6 with peak assignments are shown in Fig. 4. The three groups of characteristic peaks at 1.30–1.70 and 4.02–4.14 ppm are observed, which prove the existence of alkyl group in the molecular structure of 1,6-hexanediol. The peaks at 1.70–2.60 and 4.20–4.30 ppm are assigned to hydrogen atoms of the cyclohexane structure of 1,4-cyclohexanediol. From the ^1H NMR spectra, 1,6-hexanediol and 1,4-cyclohexanediol are both introduced into the polymer chain of the synthesized resin.

3.2. *Curing behavior*

Curing behavior is an important property in the automotive PCM because drying and curing zone in the roll coating process are much shorter than the conventional automotive process. The RPT instrument detects the curing behavior of the coatings on the steel substrate as a function of temperature. As shown in Fig. 5, the initial curing of each coating began at approximately 160 °C and continued to 160–200 °C for 15 min. The change in the frequency of each coating was similar. However, the final frequency was in the following order: EP-0 > EP-2 > EP-4 > EP-6. Generally, the final frequency is related to the stiffness of coatings, thus the result suggests that 1,4-cyclohexanediol having a cyclic structure is stiffer than other materials.

3.3. *Viscoelastic behavior*

Fig. 6 shows storage moduli as a function of temperature for stiff structures from 1,4-cyclohexanediol. The stiffness increased considerably, when the 1,4-cyclohexanediol/1,6-hexanediol ratio was increased. This tendency was in the following order: EP-6 > EP-4 > EP-2 > EP-0. The result attributed to the cyclohexane structure between ester linkages. A cyclohexane structure is a stiff chair form consisting of C—C single bonds which has a maximum rotational barrier at ±60° comparing to a linear structure from 1,6-hexanediol [11]. However, this structure is more flexible than a benzene structure because the cyclic structure consists of only σ-bond between $CH_2—CH_2$ components and can be rotated and bent [17]. This structure has high stiffness which can be restored to its original shape. According to Fig. 7, the T_g of each cured coating was shifted to a higher temperature with increasing loadings of 1,4-cyclohexanediol and it is expected that the stiffness of the coatings would increase at ambient temperature. It was found that the

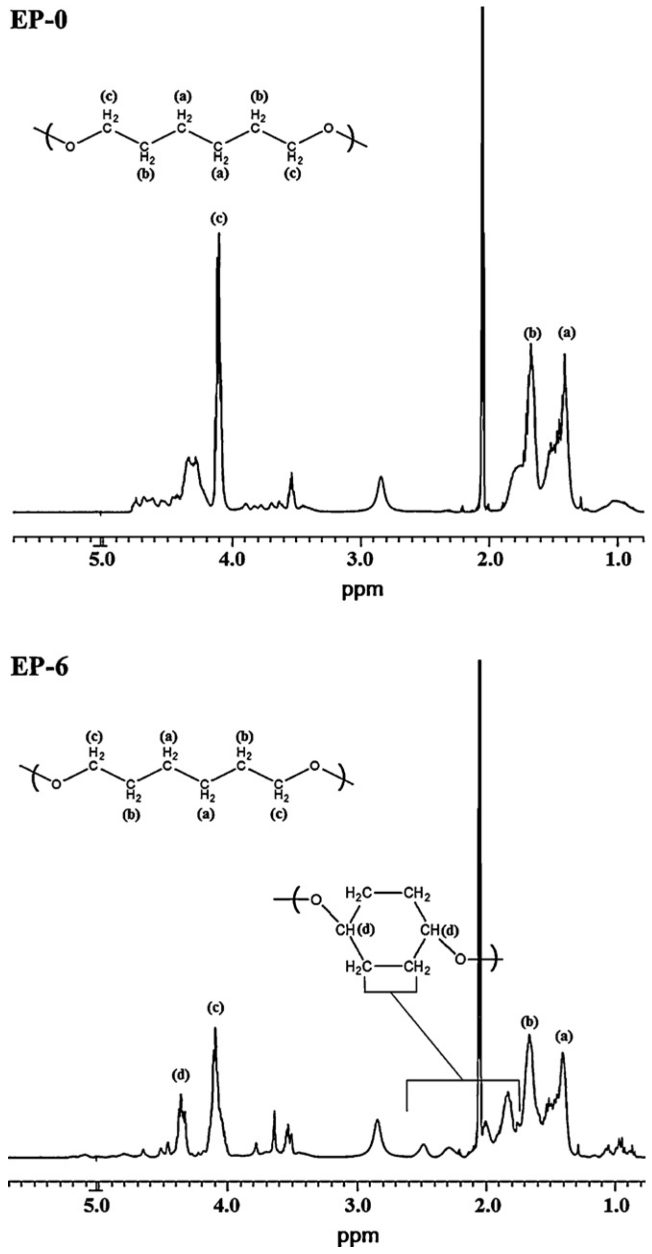


Fig. 4. ^{1}H NMR spectra of the elastomeric polyester resins.

structural shape and geometry of a polymer affect the value of T_g [18].

3.4. Physical properties

Fig. 8 shows the surface hardness of elastomeric polyester coatings with increasing 1,4-cyclohexanediol content. The T_g of an amorphous polymer is an excellent probe of its chain stiffness, which is related to its hardness [18]. For this reason, EP-6 is the highest in hardness, i.e., around 130 s and H because of the stiff structure from 1,4-cyclohexanediol. In comparison, EP-0 has the lowest hardness, i.e., around 30 s and 2B and this value is not sufficient for automotive clearcoat applications.

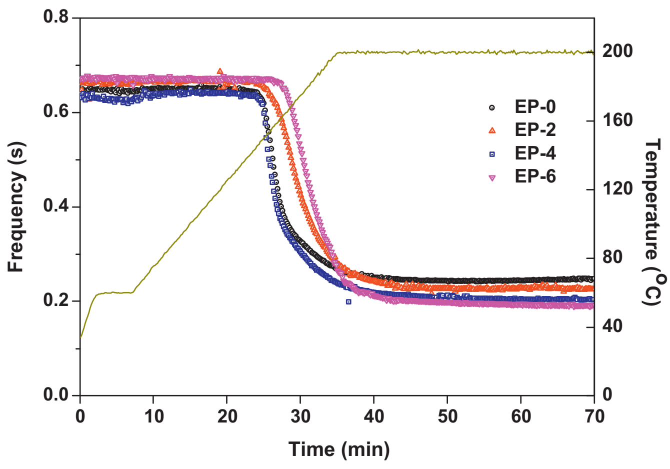


Fig. 5. Curing behavior (change of frequency(s)) of the elastomeric polyester coatings on steel substrate using a RPT instrument (data of EP-0 was derived from Ref. [9]).

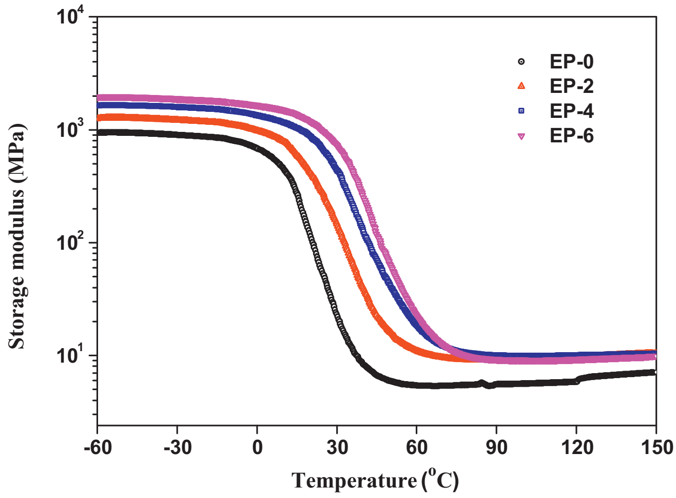


Fig. 6. Storage modulus from the DMA results of the elastomeric polyester coatings (data of EP-0 was derived from Ref. [9]).

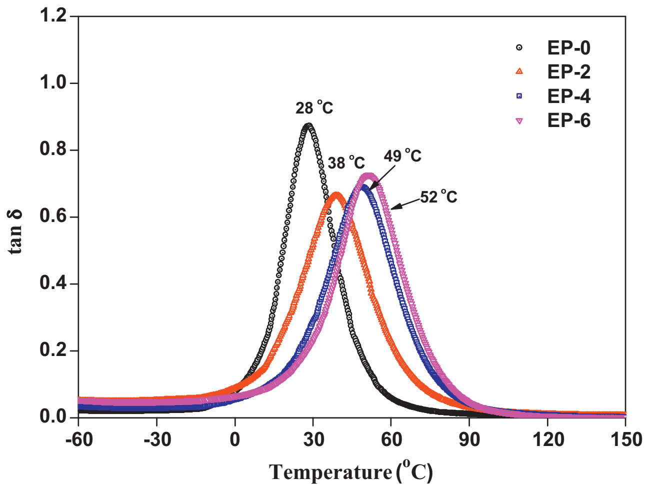


Fig. 7. tan δ from the DMA results of elastomeric polyester coatings (data of EP-0 was derived from Ref. [9]).

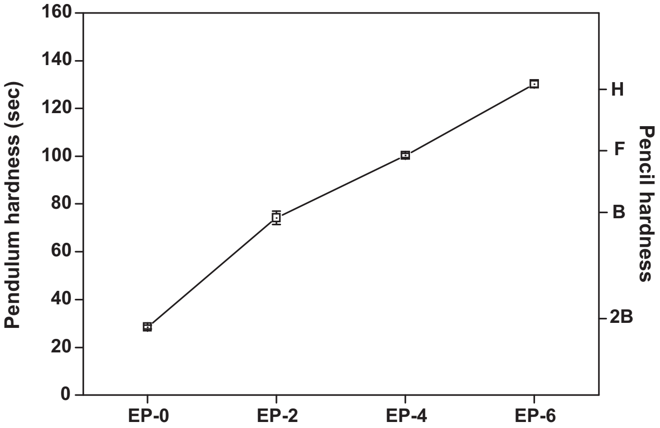


Fig. 8. Surface hardness of elastomeric polyester coatings: pendulum hardness vs. pencil hardness.

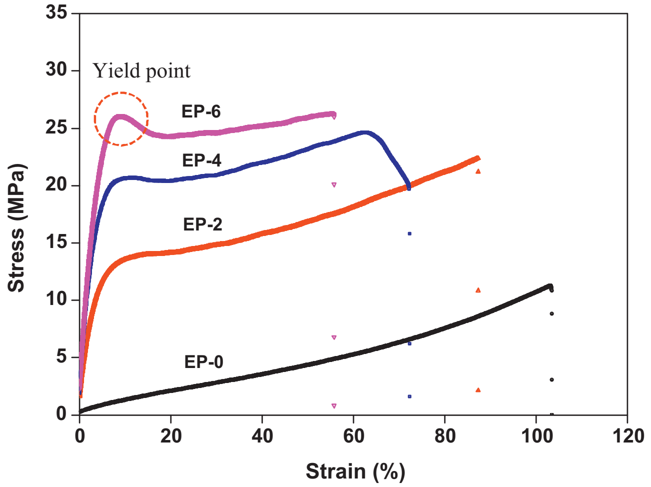


Fig. 9. Stress–strain curve of the elastomeric polyester coatings (data of EP-0 was derived from Ref. [9]).

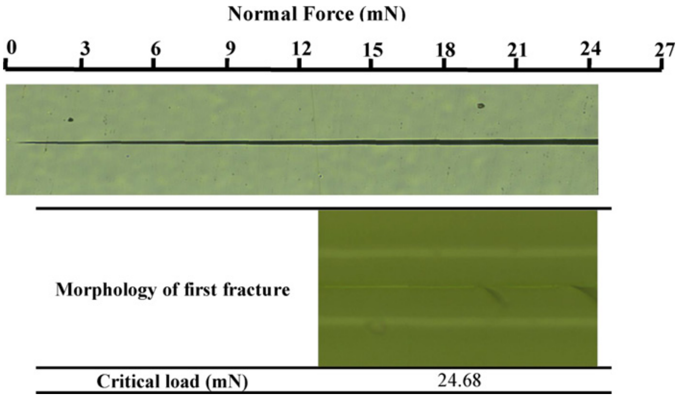


Fig. 11. Optical micrographs of the scratch paths and the first fracture made over the progressive load range (0–40 mN) on EP-6.

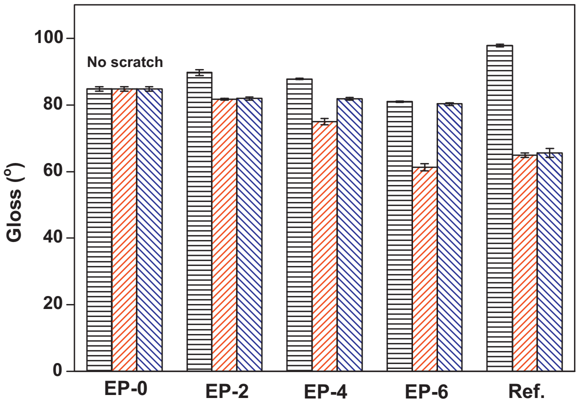


Fig. 12. Gloss change of the coatings after a car wash test and a heat treatment comparing to a conventional automotive clearcoat (100 °C, 2 min): ▤ – before a car wash test, ▨ – after a car wash test, ▨ – after a heat treatment.

3.5. Flexibility

Tensile strength and elongation are an effective factor to estimate the flexibility and stiffness of the cured coatings. Fig. 9 shows the order of the tensile strength which was similar to that of the storage modulus (EP-6 > EP-4 > EP-2 > EP-0). The stress increased with the increase in 1,4-cyclohexanediol content, and the maximum stress of EP-6 was approximately 27 MPa. In comparison, the elongation of coatings decreased with 1,4-cyclohexanediol content. However, the rate of elongation decrease was lower than that of tensile strength improvement as 1,4-cyclohexanediol content is

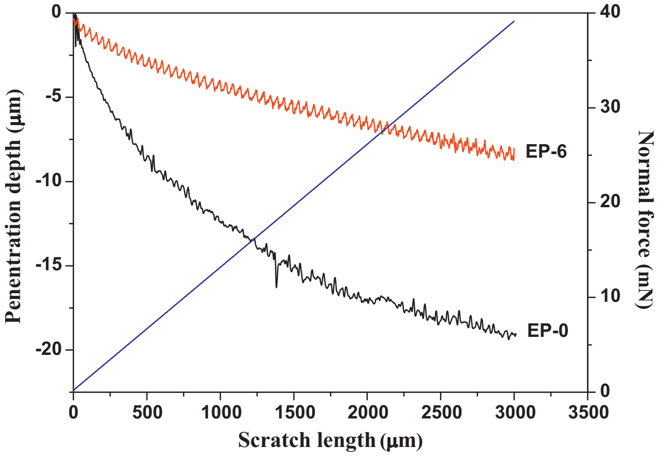


Fig. 10. Penetration depth plotted as a function of scratch length and normal force (data of EP-0 was derived from Ref. [9]).

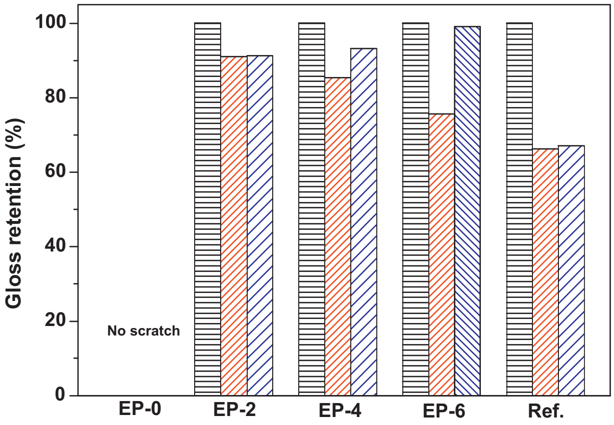


Fig. 13. Change of gloss retention after a car wash test and a heat treatment comparing to a conventional automotive clearcoat (100 °C, 2 min): ▤ – before a car wash test, ▨ – after a car wash test, ▨ – after a heat treatment.

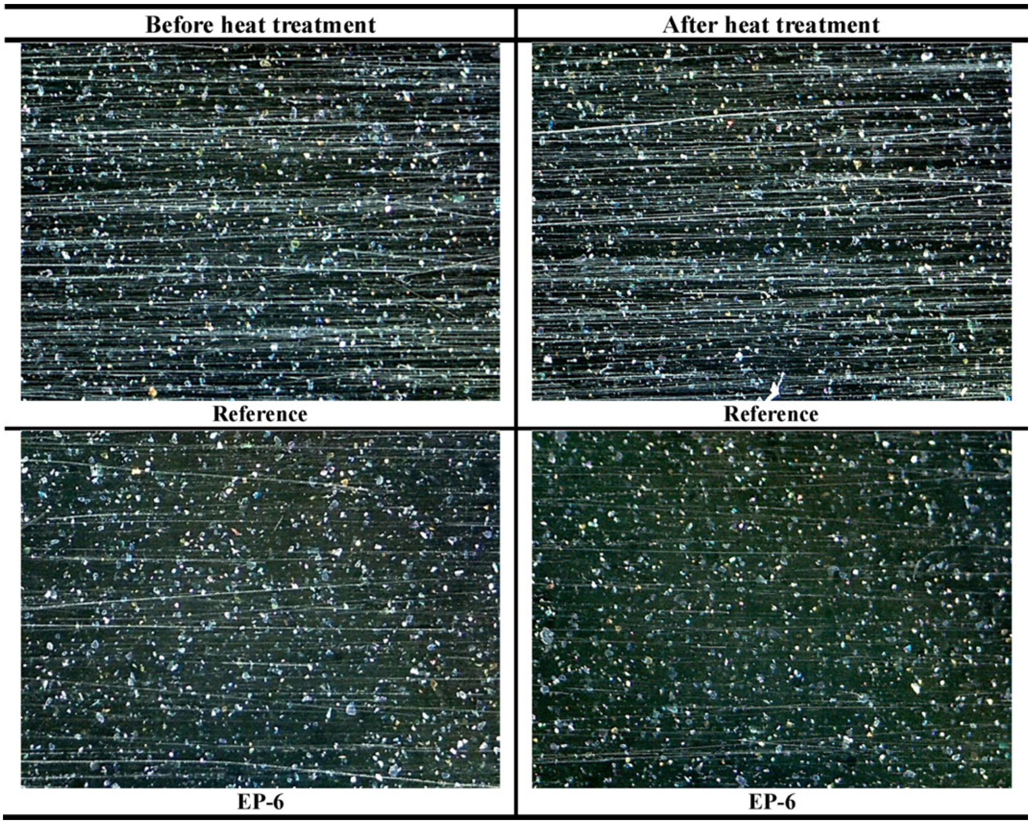


Fig. 14. Morphology of scratches on EP-6 after a car wash test and a heat treatment comparing to a conventional automotive clearcoat (100 °C, 2 min).

increased. In the case of EP-6, the elongation at break was approximately 60% which is sufficient to be used for the forming process in an automotive PCM.

In particular, EP-6 behaves as an elastic solid at the initial strain. This type of deformation is associated with the bending or stretching of the interatomic bonds between atoms of the polymer molecules, and is almost instantaneous and recoverable. Therefore, flexible polyester coatings containing a cyclic structure have both flexibility and stiffness and can be expected to reflow after scratching [19].

3.6. Scratch resistance

Scratch resistance is an important property for an automotive clearcoat and it is related to the flexibility and stiffness of the coatings. Fig. 10 shows the penetration depth of EP-0 and EP-6 using the progressive normal force mode in a nano scratch test. The greater penetration depth at the same loading means that the coating has a higher degree of softness. Results show that the penetration depth of EP-6 was lower than that of EP-0 with increasing normal force. In particular, the normal force of EP-6 was approximately 5 times larger than that of EP-0 at a scratch length of 3000 μm. In addition, the vibration of curve at EP-6 was more regular during the nano scratch analysis compared to that of EP-0. This result can be explained by the restoring force of the stiff structure from 1,4-cyclohexanediol. Fig. 11 shows the critical load at first fracture and a panoramic image of the entire path of EP-6. The critical load was approximately 25 mN, and the width of the scratch path was narrow, indicating that it is suitable for an automotive clearcoat system.

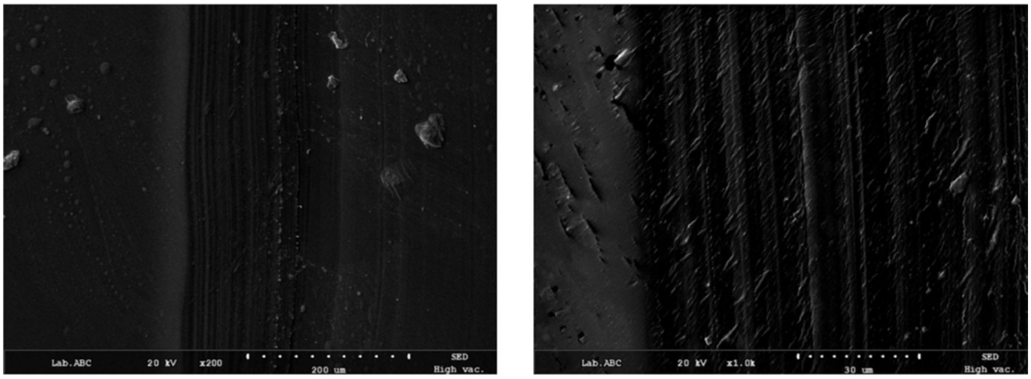
3.7. Reflow property

The reflow of elastomeric polyester coatings was determined using an Amtec laboratory car wash test. Fig. 12 shows the gloss change of coatings after a car wash test and a heat treatment. The gloss decreased after the car wash test due to the high stiffness of the cyclic structure of the increasing 1,4-cyclohexanediol. However, comparing to a conventional automotive clearcoat (Ref.), the gloss increased after heat treatment because of the reflow behavior of the cyclohexane structure of 1,4-cyclohexanediol. In addition, Fig. 13 shows the gloss retention after a car wash test and a heat treatment calculated using the following equation:

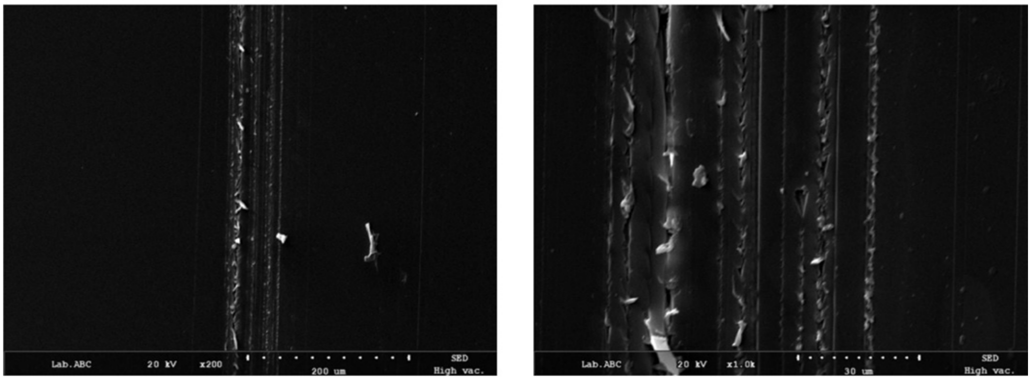
$$\text{Gloss retention}\,(\%) = \frac{G_t}{G_0} \times 100$$

where G_0 is the gloss of coating before the test and G_t is the gloss of coating after the test.

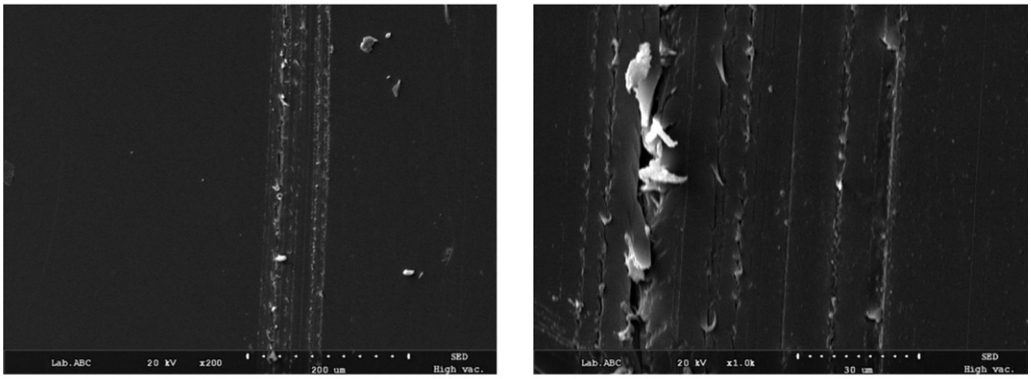
The gloss retention improved with 1,4-cyclohexanediol content after a heat treatment. In particular, the gloss retention of EP-6 after a heat treatment was approximately 100%. Fig. 14 shows morphology of scratches on coating surfaces after a car wash test and a heat treatment. In the case of EP-6, the number and depth of scratch were lower than those of reference. After a heat treatment, the scratches of EP-6 were decreased and recovered. Conversely, in case of the reference, the morphology of scratches was not altered after a heat treatment. Fig. 15 shows the morphology change of hard-damaged (using a sharp knife) surfaces after heating for different time duration. The width and depth of the scratch decreased with the increase in heating time. For 10 and 30 min heating, dents were restored, causing a decrease in the color contrast between the dents of small scratches.



EP-6, 0 min, ×200 **EP-6, 0 min, ×1000**



EP-6, 10 min, ×200 **EP-6, 10 min, ×1000**



EP-6, 30 min, ×200 **EP-6, 30 min, ×1000**

Fig. 15. Change of the damaged surface morphology with different heating time in the case of EP-6 (100 °C, 0–30 min).

4. Conclusion

We investigated an elastomeric polyester resin system to improve the scratch resistance and the reflow property of an automotive PCM. The resins were designed to increase the stiffness of coatings using 1,4-cyclohexanediol. The curing and viscoelastic behavior, physical properties, flexibility, scratch resistance and reflow behavior were measured to determine the effect of cyclic structure for an automotive PCM.

Regarding curing behavior, the synthesized polyester coatings exhibited a similar curing time and curing temperature. However, at the end of curing, the frequency was decreased with increasing 1,4-cyclohexanediol content. This suggests that 1,4-cyclohexandiol is stiffer than 1,6-hexanediol. According to DMA, the stiffness increased considerably when the 1,4-cyclohexanediol/1,6-hexanediol ratio was increased. In terms of the tan δ result, the T_g of coatings was shifted to higher temperature with 1,4-cyclohexanediol content. The surface hardness of elastomeric polyester coatings increased with increasing 1,4-cyclohexanediol content. This behavior corresponds to the increasing T_g, which is an excellent probe for chain stiffness of amorphous polymer. For tensile test results, the percent elongation decreased with increasing 1,4-cyclohexanediol content. However, the elongation at break of EP-6 was approximately 60% which is sufficient for forming process applications in an automotive PCM.

From the result of nano scratch tests, EP-6 has high scratch resistance and regular vibration of curve due to the restoring force of the stiff structure of 1,4-cyclohexanediol. Consequently, increasing gloss recovery was observed after a heat treatment following the car wash test when the content of cyclohexane structure was increased. In the case of a deep scratch, the reflow property of coating was revealed with a heat treatment. Therefore, a cyclohexane structure is a good factor for improving the scratch resistance and reflow property of polyester coatings.

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