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Test method

# Prediction of formability in drawing of PCM using tensile test and DMA creep test

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ABSTRACT

An automotive pre-coated metal system (PCM) has been investigated to avoid the wet coating process out of environmental concerns. However, automotive pre-coated metal sheets must have high formability to withstand the harsh conditions such as cutting, pressing and stamping processes. For this reason, the deep drawing test is commonly used to evaluate the formability of PCM. We have investigated and reported attempts to increase the flexibility and stiffness of polyester coatings for automotive PCM. However, it is difficult to predict whether or not the coating would form in the drawing test.

For this reason, we investigated a new method to predict formability in the drawing of PCM using a tensile test and DMA creep test. The correlation between tensile test and creep test was examined and forming coefficients $F_U$ and $F_\varepsilon$ were proposed.

Results show that if $F_U$ and $F_\varepsilon > 1$, the PCM sample would have sufficient formability under the drawing condition. However, the tensile strength of film, if below the compressive stress of the deep drawing punch or die, also determines the success of forming in the drawing test.

## 1. Introduction

Currently, environmental issues are a major concern and a problem in the automotive industry. In particular, the wet coating process discharges a mixture of water and organic/inorganic materials, whose amounts are restricted by environmental regulations. Hence, waste water purification and solvent capture are essential for this coating process.

In the pre-coated metal system (PCM), a roll coating process is used instead of wet coating; therefore problems of solvent evaporation can be eliminated. PCM is manufactured on a sheet or coil coating line and processed and assembled in factories. It has been used for household electric appliances, building materials and others. This pre-coated metal system also offers other advantages such as improved productivity and energy saving [1].

One pre-requisite for adopting the automotive pre-coated metal system is that all coatings must have high flexibility and formability to withstand harsh conditions, such as cutting and pressing processes after the metal is coated [2–6]. For this reason, the deep drawing test is commonly used to evaluate the formability of PCM. Deep drawing is one of the most important and widely used manufacturing processes for metal forming. During the drawing, a flat PCM sheet is deeply drawn by using a punch that presses a blank into a die cavity as shown in Fig. 1. To formulate a coating that will withstand these harsh conditions, we have investigated and reported several attempts for increasing flexibility and stiffness of polyester coatings. However, it is difficult to predict whether or not the coatings would form in the deep drawing test [1,7].

As tensile strain is mainly developed in a coating film in the forming process of PCMs, coating films with high elongation are favorable. In comparison, the strain behavior of the

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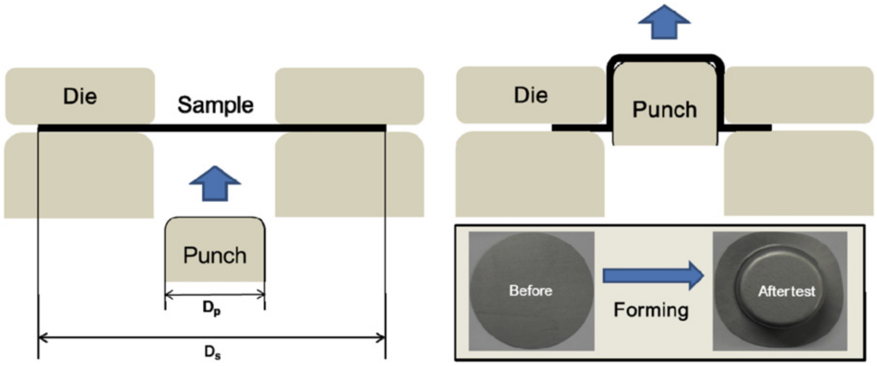

**Fig. 1.** The concept of cylindrical deep drawing test. $D_s$: diameter of sample, $D_p$: diameter of punch.

coating film in a deep drawing process is more complicated because both tensile and compressive strains are developed [8]. In a previous study, rheological properties of polyester/melamine coatings made from different molecular weight polyesters with different glass transition temperatures ($T_g$) and cross-linking densities were examined, and the formability of coating films was discussed from rheological aspects, especially creep compliance and stress relaxation [5,8]. However, only relative correlations between viscoelastic behavior and crosslink density and $T_g$ were established.

For this reason, we investigated in this study a novel method to predict formability in the drawing of PCM using the suggested parameters. From these suggestions, we defined the correlation between a tensile test and creep test using forming coefficients $F_U$ and $F_\varepsilon$. To prove our prediction. Tensile, DMA creep and cylindrical deep drawing tests were used to evaluate the formability of coatings.

## 2. Experimental

### 2.1. Sample preparation

Five types of synthesized polyester resins were prepared for automotive PCM having different values of hydroxyl number [9]. Hexamethoxymethylmelamine (HMMM, Cytec), a curing agent in the pre-coated metal system, was used. An amine type curing catalyst (NACURE 5925) was supplied by KING Industries. Xylene was used as a solvent to reduce viscosity of the synthesized resin. The polyester coatings were prepared by blending polyester resin and HMMM with the catalyst according to the hydroxyl number of polyester resin. The blended mixture was coated onto a galvanized steel sheet using a 40 μm bar applicator and cured at 150 °C for 30 min. For DMA analysis and tensile tests, the films were coated on disposable aluminum dishes and cured under the same conditions. The width and thickness of cured films were 6.5 mm and 0.5 mm, respectively [1].

### 2.2. Methods

#### 2.2.1. Investigation of parameters for prediction of formability

To study and describe different parameters of formability for automotive PCM the following notations are used:

$A_t$: Total area of PCM sample after drawing test
$A_0$: Total area of PCM sample before drawing test
$\sigma_p$: Compressive stress on PCM sample
$R_f$: Forming ratio
$U_T$: Strain energy at $R_f$ in tensile test
$U_C$: Strain energy of coating film in creep test
$F_U$ ($U_C/U_T$): Forming coefficient based on strain energy
$\varepsilon_c$: Strain at $\sigma_p$ during the creep test
$F_\varepsilon$ ($\varepsilon_c/R_f$): Forming coefficient based on strain

Generally, the punch or die pressure of the drawing machine is indicated in tons or kilograms. We recalculated the unit of die pressure from kilogram to Newton per total area of PCM sample using Eq. (1).

$$\text{Compressive stress on PCM sample}(\sigma_p) = \frac{\text{die pressure }(N)}{\text{total sample area before drawing test }(\text{mm}^2)} \tag{1}$$

As shown in Fig. 2, forming ratio $R_f$ can be calculated by measuring the size of the PCM sample before and after deep drawing. The sample is assumed to be drawn into a cup having a uniform cylindrical wall. Assuming no change in the coating film thickness after drawing,

$$\text{Forming ratio }(R_f) = \left({A_t}/{A_0} - 1\right) \times 100(\%) \tag{2}$$

#### 2.2.2. Gel permeation chromatography (GPC)

The molecular weight and polydispersity were measured using an YL9100 GPC SYSTEM (Young Lin, Korea) apparatus consisting of 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. The molecular weight and polydispersity of the synthesized resins are listed in Table 1.

#### 2.2.3. Deep drawing test

A cylindrical deep drawing test was performed to examine the formability of the coated metal sheet on an automotive assembly line as shown in Fig. 1. The punch size

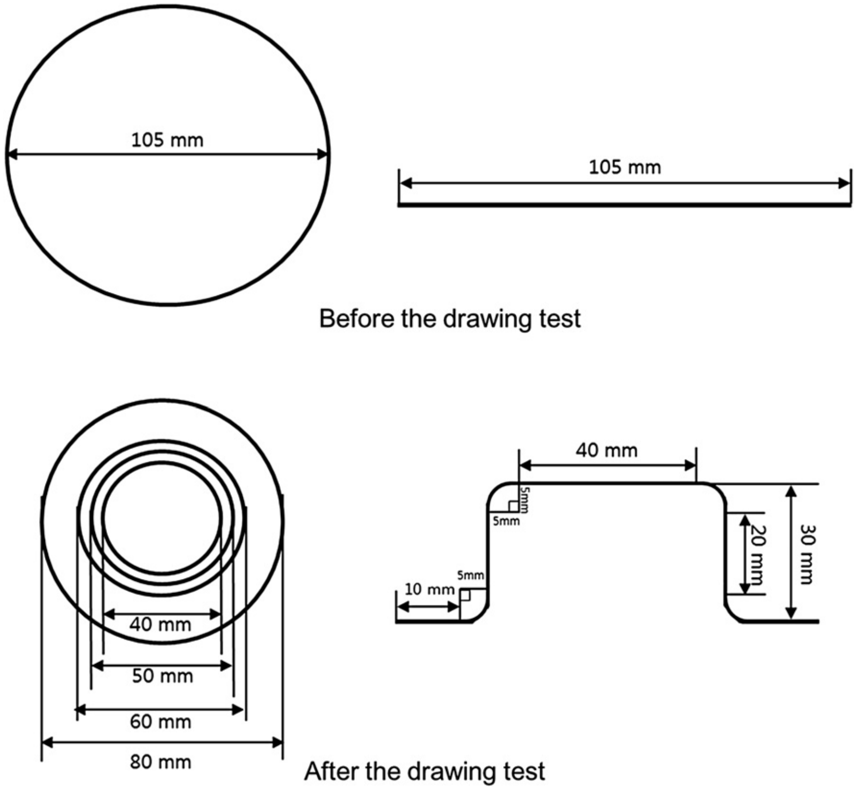

**Fig. 2.** Schematic diagram of samples in cylindrical drawing test.

of the deep drawing was 40 diameter (mm) and the temperature of the drawing die was 25 °C. The speed and pressure of the deep drawing machine were 20 mm/min and 4500 kg for 1.5 min. The integrity of the coating film after deep drawing was evaluated by observing the appearance of the formed parts. Table 2 shows the conditions of the cylindrical deep drawing test for evaluating formability [8].

### *2.2.4. Dynamic mechanical analysis (DMA)*

Dynamic mechanical analysis (DMA) was performed using a Dynamic Mechanical Analyzer Q-800 (TA Instruments, USA). The machine was used to determine glass transition temperature and crosslink density. The crosslink density ($\upsilon_c$) was derived from the minimum storage modulus ($E'_{min}$) and temperature at minimum storage modulus ($T_{E'_{min}}$) in the rubbery plateau region. The crosslink density was calculated using the following equation [1,6]:

$$\upsilon_c = \frac{E'_{min}}{3RT_{E'_{min}}} \qquad (3)$$

The test was performed in a tensile mode under the following conditions: a frequency of 1 Hz, strain of 0.3% and temperature from −60 °C to 160 °C at a heating rate of 2 °C/min.

A DMA creep test can evaluate not only the creep compliance of polymer, but also the strain energy and elongation under the constant stress condition. This test

**Table 1**
Characterization of the polyester resin for PCM.

| Property | PE-A[a] | PE-B[a] | PE-C[a] | PE-D[a] | PE-E |
|---|---|---|---|---|---|
| Number average molecular weight ($M_n$) | 4200 | 4470 | 4770 | 4900 | 4500 |
| $n_{OH}$ (mg KOH/g)[b] | 43 | 60 | 78 | 95 | 57 |
| $T_g$ (°C) | 30.5 | 38.8 | 46.1 | 51.9 | 44.2 |
| Crosslink density ($10^{-3}$ mol/cm$^3$) | 0.2 | 0.4 | 1.1 | 1.5 | 0.5 |
| Curing agent (mol)[c] | 1.10 | 1.55 | 2.00 | 2.45 | 1.44 |

[a] Data of PE-A, PE-B, PE-C and PE-D were from Ref. [9].
[b] $n_{OH}$ – Theoretical hydroxyl number of polyester resins.
[c] Content of curing agent was calculated based on hydroxyl number of polyester resins.

**Table 2**
Conditions of the cylindrical deep drawing test.

| | Conditions |
|---|---|
| Shape of punch | Cylinder |
| Shoulder radius of punch (mm) | 5 |
| Shoulder radius of die (mm) | 5 |
| Size of punch ($D_P$, mm ϕ) | 40 |
| Size of PCM sheet ($D_S$, mm ϕ) | 105 |
| Drawing height (mm) | 30 |
| Punch & die Pressure (kg) | 4500 |
| Punch speed (mm/min) | 20 |

**Table 3**
Change of total sample area after deep drawing test.

| Before deep drawing test ($mm^2$) | After deep drawing test ($mm^2$) | Forming ratio ($R_f$) |
|---|---|---|
| 2756 π | 3400 π | 23.36% |

was used to reproduce the deformation of films under same conditions of the deep drawing test: stress of 5 MPa for 1.5 min at 25 °C.

### 2.2.5. *Tensile strength*

The flexibility of the polyester coatings was determined from tensile tests using a universal testing machine (UTM, Zwick GmbH.) with rectangle-shaped specimens according to the ISO 527-3 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 section 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 (%) [1,10].

## 3. Results & discussion

### 3.1. *Characterization of polyester resin*

Table 1 lists the characteristics of the polyester resins including molecular weight, hydroxyl number, $T_g$ and crosslink density ($\upsilon_c$). To define factors of drawing, molecular weights of resins were fixed in a narrow range of 4000–5000 g/mol. In addition, the $T_g$ of resins was targeted to be above the drawing temperature (25 °C) to prevent effects of softening which occurs above $T_g$.

### 3.2. *Calculation of parameters of formability*

According to Eq. (1), $\sigma_p$, the compressive stress on a PCM sample is 5 MPa in the deep drawing test. Table 3 shows $R_f$, the forming ratio calculated according to Eq. (2). This value refers to the degree of elongation of the coated film on a steel substrate during the drawing test. This $R_f$ value, together with $\sigma_p$, were used to set the test conditions for the tensile and creep tests used in this study.

### 3.3. *Deep drawing*

Fig. 3 shows the PE series cylindrical cups formed under the conditions listed in Table 2. After 30 mm drawing, the side of the formed cup was torn, except those of PE-B and E. In the case of PE-A, the film was damaged and delaminated by the die pressure because its tensile strength is below 5 MPa, as shown in Fig. 4. For PE-C and D, large defects occurred attributable to the increasing crosslink density [8,9].

### 3.4. *Relationship between flexibility in tensile test and creep behavior*

### 3.4.1. *Strain energy from tensile test*

Fig. 4 shows the stress-strain behavior of coating films during tensile tests. To define the relationship between flexibility and creep behavior, the parameter $U_T$ is suggested as shown in Fig. 5 and can be calculated using Eq. (4).

$$\text{Strain energy at } R_f \text{ in tensile test}(U_T) = \int_0^{R_f} \text{strain} - \text{dependent stress curve } d\varepsilon \quad (4)$$

The value of $U_T$ indicates the pure strain energy of film to form at $R_f$ in the stress-strain curve during drawing.

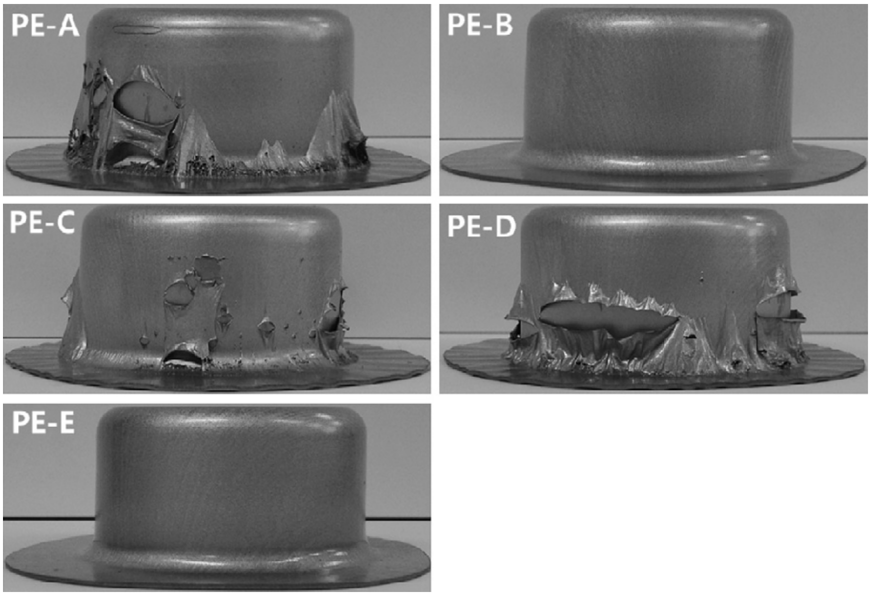

**Fig. 3.** Formability of PE series on GI substrate using a cylindrical deep drawing tester (data of PE-A, PE-B, PE-C and PE-D were from Ref. [9]).

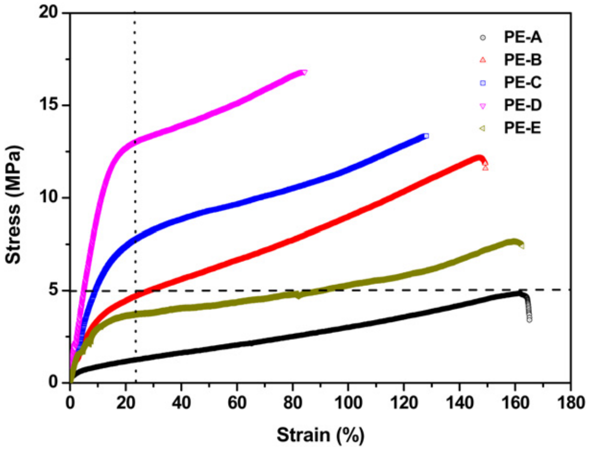

**Fig. 4.** Stress-Strain curve of the PE series – vertical dot line at 23.36%: represents forming ratio ($R_f$), horizontal dash line at 5 MPa: compressive stress during the drawing test (data of PE-A, PE-B, PE-C and PE-D were from Ref. [9]).

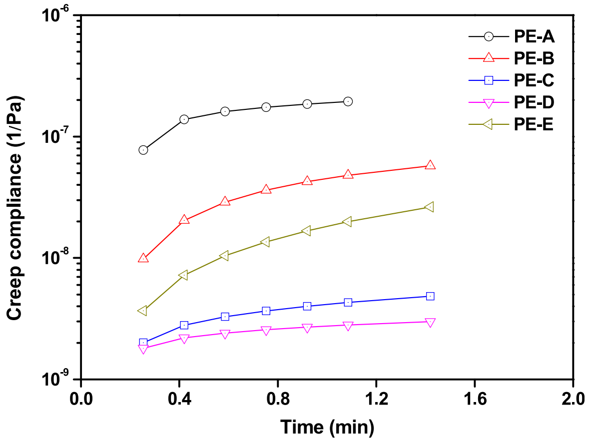

**Fig. 6.** Time-dependent creep compliance of polyester coatings with different crosslink density in creep test (PE-A: elongation breaking at 1 min).

### *3.4.2. Strain energy from creep test*

Fig. 6 shows the creep compliance of PE series at $\sigma_p$ of 5 MPa. With the increase of crosslink density, the creep compliance decreased accordingly. This tendency was in the following order: PE-A > PE-B > PE-E > PE-C > PE-D. This result is attributed to the stiffer film with increasing crosslink density. However, the usefulness of creep compliance is limited to evaluating the relative flexibility between samples.

For this reason, the concept of $U_C$ is suggested as shown in Fig. 7, calculated using the following equation:

$$\text{Strain energy of coatings in creep test } (U_C) = \int \text{strain} - \text{dependent stress curve } d\varepsilon \qquad (5)$$

The stress-strain value is exported from the DMA creep data. The value of $U_C$ indicates the strain energy of coating film to form at $\sigma_p$ (5 MPa) for drawing time (1.5 min). In addition, $\varepsilon_{c,}$ i.e., the strain at $\sigma_p$ during the creep test, is suggested for comparing to $R_f$ from the deep drawing test.

### *3.4.3. Relationship between flexibility in tensile test and creep behavior*

According to the suggested parameters, it can be assumed that, if the value of $U_C$ is larger than that of $U_T$, the PCM sample can be formed under the drawing condition because the capacity of elongation would be favorably larger than $R_f$. From this hypothesis, we propose a forming coefficient based on stain energy, $F_U$ as expressed in Eq. (6)

$$\text{Forming coefficient based on strain energy } (F_U) = U_C/U_T \qquad (6)$$

If $F_U > 1$, the PCM sample would have sufficient formability at the drawing condition with 5 MPa of punch or die pressure (Fig. 8).

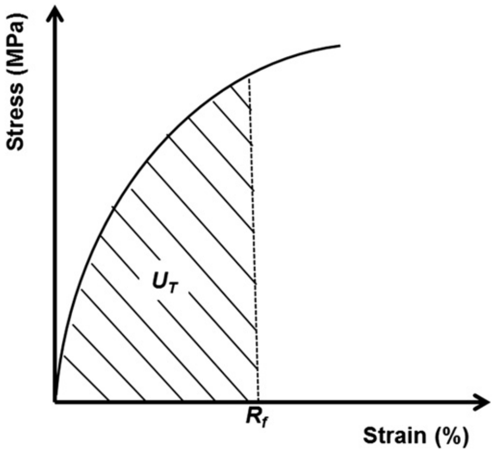

**Fig. 5.** Strain energy at $R_f$ in tensile test ($U_T$).

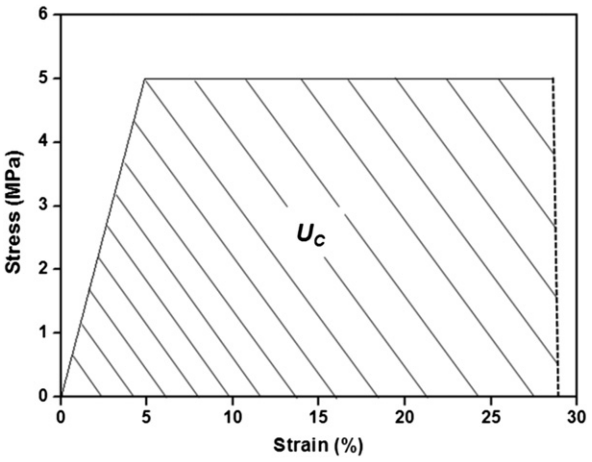

**Fig. 7.** Strain energy of coating film ($U_C$) in creep test (5 MPa, 1.5 min).

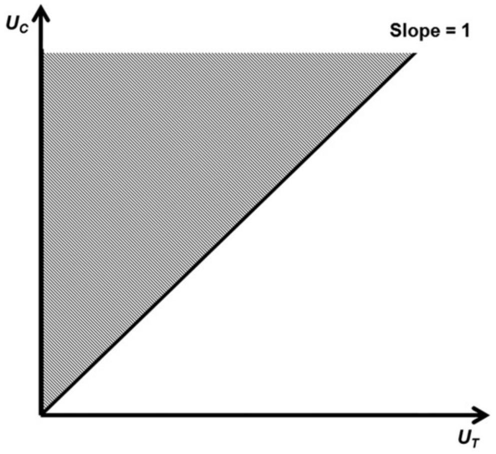

**Fig. 8.** Relationship between $U_T$ and $U_{C.}$

**Table 4**
Calculated values as a function of parameter.

| Sample | $U_T$ | $U_C$ | $F_U$ ($U_C/U_T$) | $\varepsilon_c$ (%) | $F_\varepsilon$ ($\varepsilon_c/R_f$) |
|---|---|---|---|---|---|
| PE-A[a] | 20.63 | 388.80 | 18.58 | 96.80 | 4.15 |
| PE-B | 76.46 | 131.17 | 1.72 | 28.67 | 1.23 |
| PE-C | 120.67 | 9.68 | 0.08 | 2.45 | 0.10 |
| PE-D | 205.57 | 5.32 | 0.03 | 1.52 | 0.07 |
| PE-E | 64.26 | 114.76 | 1.79 | 24.15 | 1.03 |

$U_T$: strain energy at $R_f$ in tensile test.
$U_C$: strain energy of coating film in creep test.
$F_U$ ($U_C/U_T$): forming coefficient based on strain energy.
$\varepsilon_c$: strain at $\sigma_p$ during the creep test.
$F_\varepsilon$ ($\varepsilon_c/R_f$): forming coefficient based on strain.
[a] PE-A: elongation breaking at 1 min in creep test.

In addition, a forming coefficient based on strain, is also proposed according to Eq. (7)

$$\text{Forming coefficient based on strain } (F_\varepsilon) = \varepsilon_c/R_f \qquad (7)$$

where the numerator of the ratio indicates the elongation percentage of film in creep tests at $\sigma_p$ (5 MPa) for drawing time (1.5 min), the denominator indicates the forming ratio during the drawing test. This expression means that if $F_\varepsilon > 1$, the PCM sample would have sufficient formability at the drawing condition with 30 mm of drawing height. Table 4 lists the calculated values of the parameters for different samples. In the case of PE-A, B, and E, the parameters of $F_U$ and $F_\varepsilon$ are over 1 and the forming coefficients of other samples are below 1. According to the results, we can predict that PE-B and E could be formed by drawing but not PE-A because of its failure (at 1 min) before the completion of the creep test. In comparison, PE-C and D, which have high crosslink density, exhibit lower strain at 5 MPa than $R_f$, the forming ratio, as shown in Fig. 9. In contrast, the strain at 5 MPa was lower than $R_f$ for PE-C and D, which were observed to have large forming defects, corresponding to their high crosslink density.

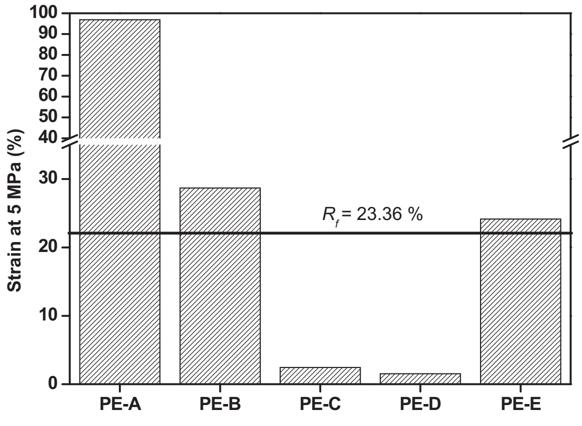

**Fig. 9.** Strain at 5 MPa (%) of PE series during creep test (PE-A: elongation breaking at 96.89% before the end of test).

## 4. Conclusions

The formability of polyester coatings was studied with different hydroxyl number and the forming coefficients $F_U$ and $F_\varepsilon$ were investigated. To define factors of drawing, molecular weight of resins were fixed in a narrow range of 4000–5000 g/mol. In addition, the $T_g$ of resins were targeted to be above the drawing temperature (25 °C) to prevent effects of softening which occur above $T_g$. To prove usefulness of these parameters, cylindrical deep drawing, tensile and DMA creep tests were performed to evaluate the formability of coatings.

According to the results, we can predict that PE-B and E could be formed by drawing but not PE-A because of its failure (at 1 min) before the completion of the creep test. In comparison, PE-C and D, which have high crosslink density, exhibit lower strain at 5 MPa than $R_f$, the forming ratio. In contrast, the strain at 5 MPa was lower than $R_f$ for PE-C and D, which were observed to have large forming defects, corresponding to their high crosslink density.

Results show the following conclusions:

(1) The tensile strength of film must be above the compressive stress ($\sigma_p$) of the deep drawing punch or die.

(2) If $F_U$ and $F_\varepsilon > 1$, the PCM sample would have sufficient formability under the drawing conditions.

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