

The Characteristic of Epoxy Resin That Makes Decomposition Easy by Blending Thermoplastic Polymer

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ABSTRACT: To recover and reuse valuable metal materials from waste at the time of abandonment, a thermosetting resin (epoxy resin) easily disassembled with solvent treatment was developed by blending a thermoplastic polymer (polyethersulfone, PES) to the thermosetting resin. The influence of the morphology of the cured resin upon the decomposition nature by the organic solvent was investigated. In the unmodified resin and the modified resin with homogeneous morphology, the cured resins did not decompose easily when they were dipped in dimethylformamide (DMF) at 23°C. It took 13 days or more to recover the metal materials molded into the cured resin. On the other hand, in the modified resin with continuous-phase morphology of the thermoplastic polymer, decomposition proceeded rap-

idly. The resin portion was decomposed completely in only 2 days and the molded metal materials were recoverable. Furthermore, the material with a continuous variation in the phase morphology of the cured resin, that is, continuously variable morphology material, was created by attaching a slope to the molding temperature of a sample. A place to decompose can be selected by using this material, which allows for easy recovery of molded valuables at the time of abandonment. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 89: 527–534, 2003

Key words: alloys; thermosets; morphology; phase separation; recycling

INTRODUCTION

While the consciousness of environmental protection increases globally, consideration for the environment in both electricity and electronic products is becoming indispensable. An epoxy resin compound with superior electric insulation and heat resistance is widely used for a mold resin of electronic products such as integrated circuits. Reclamation disposal of most products that consist of thermosetting resin such as epoxy resin is carried out without reuse because it is insoluble to solvent or heat. However, the amount of abandoned materials increases each year. To reduce the environmental load by the effective use of resources and reclamation, development of practical recycling technology is strongly desired.

In electricity and electronic products, expensive valuable metals are molded by a thermosetting resin such as an epoxy resin. To recover and reuse these valuable metals from waste, development of a thermosetting resin that may be easily disassembled by external stimulus, etc., is needed.

Therefore, we tried to develop a thermosetting resin disassembled easily by blending a thermoplas-

tic polymer. The blend of the thermoplastic polymer to the thermosetting resin has been undertaken to improve the performance of the thermosetting resin.^{1–39} The morphology of the cured resin is very important in such a combination. The two-phase morphology is established via reaction-induced spinodal decomposition in such combination.⁹ That is, starting from a homogeneous mixture, the system is thrust into a two-phase region by the increase in molecular weight of the thermosetting resin at the early stage of curing and spinodal decomposition takes place to provide regularly phase-separated structure. However, the morphology is frozen when the glass transition temperature (T_g) exceeds the curing temperature. The final morphology is a result of the competition between the molecular weight increase of the thermosetting resin, leading to the phase separation, and the simultaneous crosslinking which suppresses it. Therefore, many attempts have been made to control the morphology of the thermoplastic/thermoset system.^{21,29,31,32}

We also added the thermoplastic polymer (polyethersulfone, PES) to the thermosetting resin (i.e., epoxy resin) to improve heat resistance and toughness of a cured resin.¹⁵ The phase morphology of the cured resin was controllable by changing the molding conditions. Therefore, this technology was applied to the development of an easy decomposition-type thermosetting resin.

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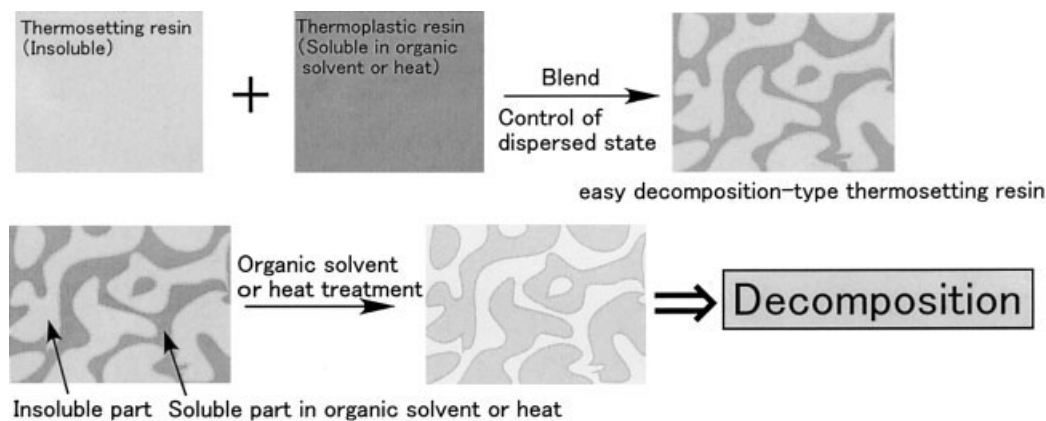
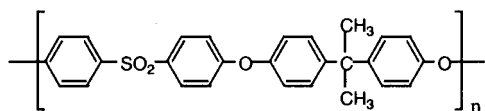


Figure 1 Concept of easy decomposition-type thermosetting resin.

EXPERIMENTAL

Materials and preparation of samples

The epoxy resin used in this work was diglycidyl ether of the biphenyl-type (YX4000; Japan Epoxy Resins Co., Ltd.). The curing agent was a phenol novolak-type resin (PSM4261; Gun Ei Chemical Industry Co., Ltd.), and the accelerator was triphenylphosphine (TPP). As a thermoplastic polymer, the engineering plastic, PES, (Teijin Amoko Engineering Plastic) was used. Its structure is given as:



The concentration of the added PES varied from 0 to 20% by weight. The epoxy resin was compounded stoichiometrically with a curing agent: one epoxy group corresponds to one hydroxyl group of the curing agent. The compound was stirred at 135°C until the PES dissolved homogeneously in a mixture of the epoxy resin and curing agent. The TPP was then added, and the mixture was rapidly removed from the apparatus. The mixture was molded at 140, 160, or 180°C for 10 min with a transfer press (Kohtaki Inc.) and then cured at 175°C for 7.5 h.

Measurements

The morphology of the fracture surfaces was observed by using a scanning electron microscope (SEM: JSM-T20, JEOL, Co., Ltd.). The fracture surfaces were coated with a gold layer about 200-Å thick.

The adhesion properties of the encapsulants to the Fe-Ni 42 alloy were measured by pulling out the specimen. In the adhesion examination, the specimen-embedded metal (5 × 20 × 0.5 mm) 5 mm in the resin part (5 × 10 × 15 mm) was used as shown in Figures

5–7. The metal portion of the specimen was pulled at the rate of 1 mm/min.

The heat resistance (T_g 's) of the cured epoxy resins was measured by a dynamic mechanical analyser (DMA) by using a nonresonance forced vibration viscoelastometer (RDA, Rheometrics Co., Ltd.). The frequency was adjusted to 1 Hz, and the heating rate was 2°C/min in air.

The fracture toughness of the cured epoxy resins was evaluated from the critical value K_{Ic} of the stress intensity factor for the initiation of crack growth, which was determined from three-point bent specimens (5 × 15 × 75 mm with a span of 59 mm) according to ASTM E399-78. Careful tapping with a fresh razor blade formed a sharp precrack. The specimen was loaded at a constant crosshead speed (0.5 mm/min). Five specimens were tested for each sample.

The mechanical properties of the cured epoxy resins were determined with a Shimadzu autograph AG-5000 D universal testing machine. Flexural tests were carried out at a crosshead speed of 1.5 mm/min according to JIS-K 6911. Bars of 3 × 10 × 85 mm were used at a span of 48 mm to measure the flexural properties. Five specimens were tested for each sample.

RESULTS AND DISCUSSION

Control of the phase morphology of the epoxy resin blended with the thermoplastic polymer

The concept of an easy decomposition-type thermosetting resin is shown in Figure 1. Thermoplastic polymer that can be dissolved in organic solvent or heat is blended to a thermosetting resin, and the phase morphology of a cured resin is controlled so that the thermoplastic polymer forms a continuous phase. As mentioned above, this technology^{9,14–19,37,39} was originally successfully used for improvement of the heat

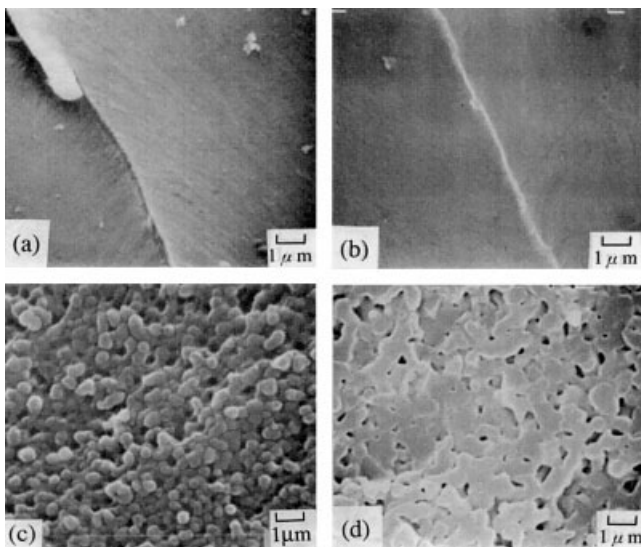


Figure 2 Scanning electron micrographs of the cured epoxy resins containing PES. Sample: (a) unmodified resin, (b–d) cured epoxy resins containing 20 wt % of PES. Molding temperature: (a) 160°C, (b) 140°C, (c) 160°C, (d) 180°C.

resistance and toughness of the cured resin. At the time of abandonment, an attempt was made to comparatively easily disassemble the thermosetting resin by dissolving the thermoplastic polymer portion with a chemical treatment or heat treatment and to recover/reuse the valuable metal materials molded into the thermosetting resin. However, to do so, a thermoplastic polymer without reaction with the thermosetting resin must be selected, because the thermoplastic polymer cannot dissolve easily by either chemical treatment or heat treatment when it reacts with the thermosetting resin. The above-mentioned model was verified in this article by using an epoxy resin that has been widely used in the electronics industry as the thermosetting resin and a PES without reaction with the epoxy resin as the thermoplastic polymer.

It is well known that the combination of epoxy resin and PES exhibits lower critical solution temperature (LCST) phase behavior.⁹ In a combination of the polymer with LCST, inclusion of a higher PES content (in the epoxy-rich composition) or a higher curing temperature gives rise to further progress of the phase separation of PES from the epoxy matrix. The morphologies of the cured resins when fixing the content of the PES to 20 wt % and changing the molding temperature to 140, 160, and 180°C were observed. The results are shown in Figure 2. In these pictures, the PES was removed by extracting the samples with dichloromethane prior to the SEM examination. The unmodified resin, to which PES was not added, showed a homogeneous morphology [Fig. 2(a)]. For the system modified with PES, the cured resin molded at 140°C exhibited a homogeneous morphology [Fig. 2(b)] analogous to the unmodified resin. In the cured

epoxy resin molded at 160°C [Fig. 2(c)], the PES separated from the epoxy matrix and formed a well-defined, dispersed phase the diameter of which was about 0.3 μm . In the cured epoxy resin molded at 180°C [Fig. 2(d)], furthermore, it was observed that the mutual PES phase was connected and the PES formed a co-continuous phase morphology composed of the epoxy matrix and PES. This indicated that the phase morphology of the cured resin was controllable by changing the molding temperature even if it had the same content of PES.

The SEM photographs of the cured resin when fixing the molding temperature at 180°C and changing the content of PES to 10 and 20 wt % are shown in Figure 3. If the content of PES was as small as 10 wt %, the cured resin formed a spherical dispersed phase of PES even if the molding temperature was as high as 180°C.

To make the continuous phase of PES form in the epoxy matrix, as mentioned above, a molding temperature of $\geq 180^\circ\text{C}$ and a PES content of ≥ 20 wt % were required. Henceforth, the characteristic was compared in the following three systems: the unmodified resin, the cured resin with a homogeneous morphology (PES content: 20 wt %; molding temperature: 140°C), and the cured resin with a continuous morphology of PES (PES content: 20 wt %; molding temperature: 180°C).

Evaluation of the decomposition nature of the thermosetting resin blended with the thermoplastic polymer

The PES of the thermoplastic polymer used here is dissolved in special organic solvents, such as dichlo-

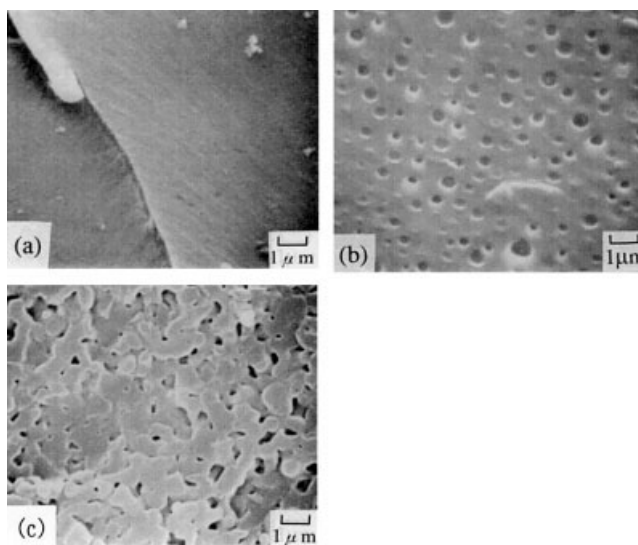


Figure 3 Scanning electron micrographs of the cured epoxy resins containing PES. Sample: (a) unmodified resin, (b–c) cured epoxy resins containing PES. Molding temperature: (a) 160°C, (b–c) 180°C. PES content: (a) 0 wt %, (b) 10 wt %, (c) 20 wt %.

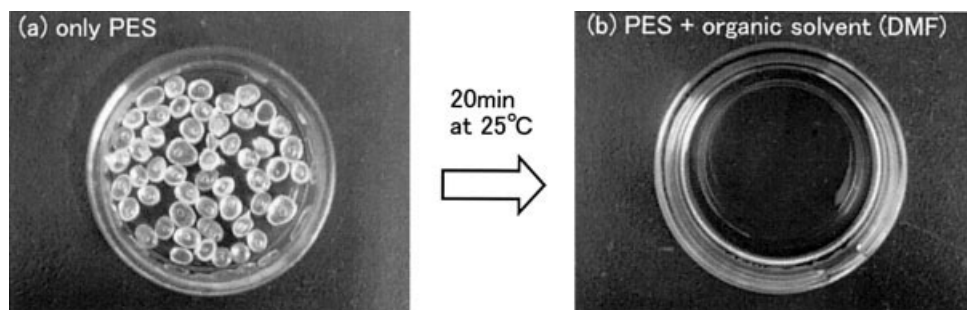


Figure 4 Solubility of PES to the organic solvent DMF.

romethane and dimethylformamide (DMF). The solubility of PES to the organic solvent DMF is shown in Figure 4. When DMF was added and about 20 min passed at 23°C, the PES was dissolved completely. The cured resin modified with PES was then treated with the DMF, and the decomposition nature was evaluated. The relation between treatment time and strength was investigated by using a piece of pull-out adhesion examination.

The change in the piece of the adhesion examination in the unmodified resin after solvent treatment is shown in Figure 5. In the unmodified resin, even if it was treated with the DMF solvent for 5 days at 23°C, there was almost no change in appearance. When treated for 10 days [Fig. 5(b)], the outside of the resin part began to disappear. When 20 days had passed from the treatment start [Fig. 5(c)], the resin portion had decomposed considerably. However, metal sub-

strates remained in the state in which they had been molded by the epoxy resin. To make the cured resin portion decompose and to recover the metal substrates from it, 25 days were required [Fig. 5(d)]. It was observed that the resin portion decomposed into a comparatively large fragment. Thus, to recover metal substrates from the unmodified resin, a long period is required from the treatment start.

The change in the PES-modified resin with the homogeneous morphology after solvent treatment is shown in Figure 6. The photograph shown in Figure 6(a) is a photograph of a piece of the adhesion examination before solvent treatment. Because PES dissolved in the epoxy matrix and a clear cured resin was formed in this system, the metal substrates molded in the cured resin portion were observed vividly in analogy with the unmodified resin. When treated for 3 days [Fig. 6(b)], the outside

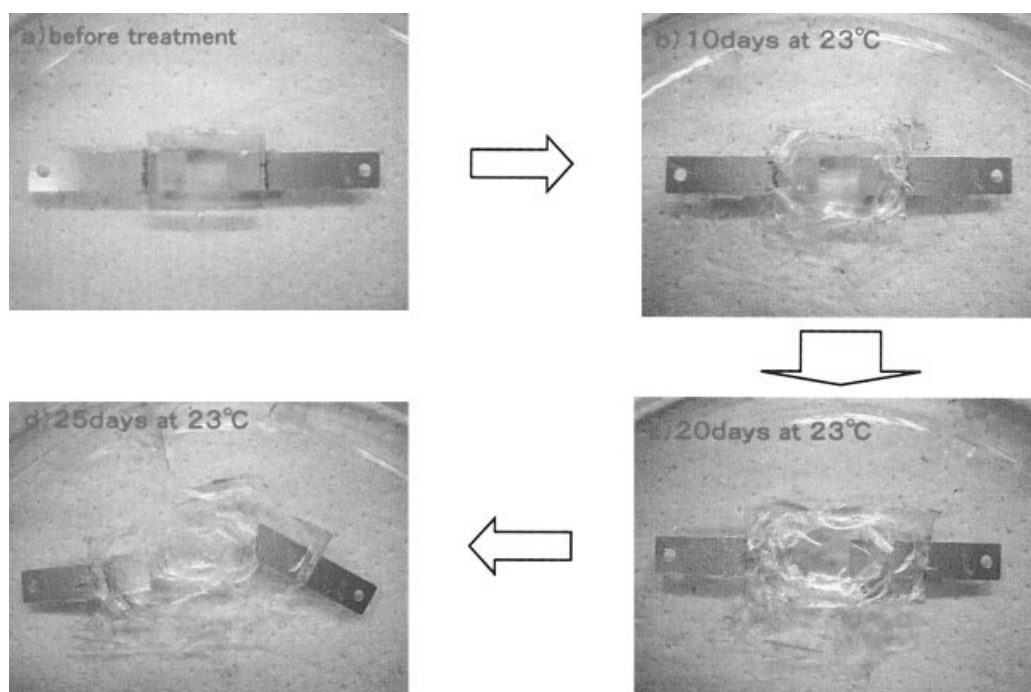


Figure 5 The change in the piece of adhesion examination in the unmodified resin after solvent treatment. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

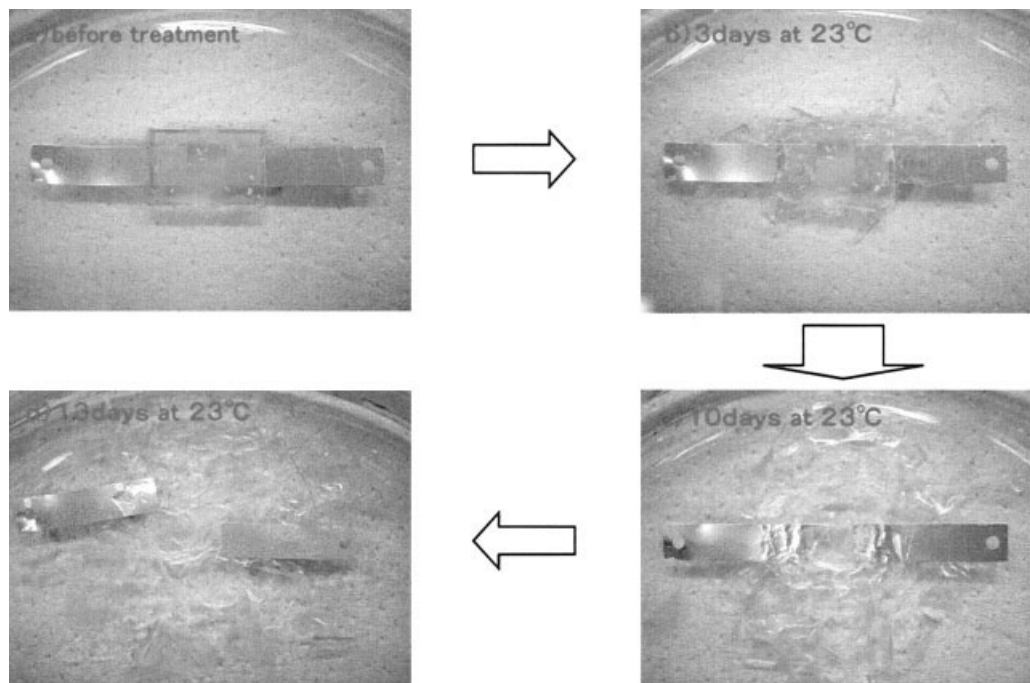


Figure 6 The change in the piece of adhesion examination in the modified resin with the homogeneous morphology after solvent treatment. Sample: PES content: 20 wt %; Molding temperature: 140°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

of the resin part began to disappear. When 10 days had passed from the treatment start [Fig. 6(c)], decomposition of the resin portion had progressed considerably. However, sufficient decomposition

does not occur until the molded substrates are recoverable. Thirteen days were required to recover the metal substrates from the piece of adhesion examination [Fig. 6(d)]. It was observed that the

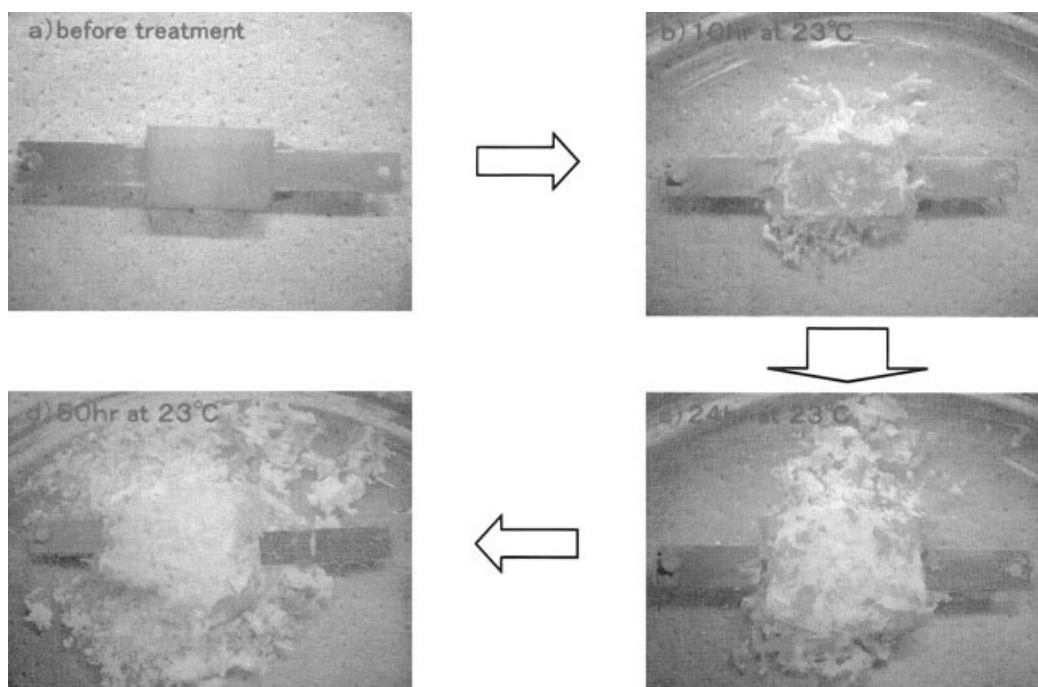


Figure 7 The change in the piece of adhesion examination in the modified resin with the continuous-phase morphology of PES after solvent treatment. Sample: PES content: 20 wt %; Molding temperature: 180°C. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

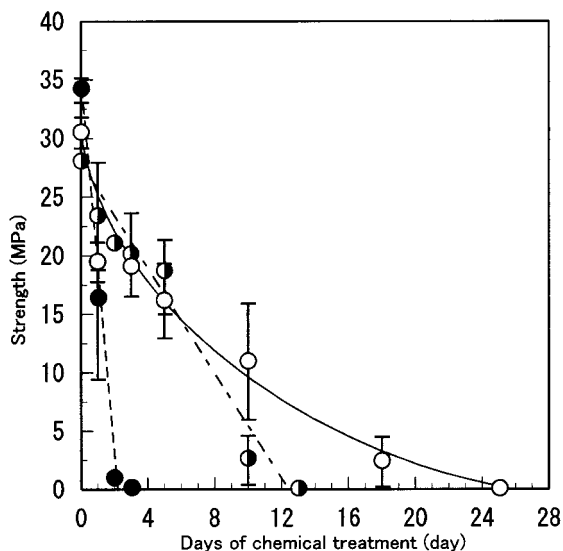


Figure 8 Decomposition nature of the cured epoxy resins containing PES by chemical treatment. Sample: ○ unmodified resin, ◐ ●, cured epoxy resins containing 20 wt % of PES. Molding temperature: ◐ 140°C; ○ 160°C; ● 180°C.

resin portion decomposed into a comparatively large fragment, analogous to the unmodified resin. Thus, although the treatment dates are shorter than that of the unmodified resin, a long time is still necessary to recover metal substrates from the PES-modified resin with homogeneous morphology.

The change in the modified resin with continuous-phase morphology of PES after solvent treatment is shown in Figure 7. The photograph in Figure 7(a) shows that the cured resin is opaque because the phase separation of PES has occurred. In this system, it decomposed from the outside of the resin part for 10 h after the treatment start [Fig. 7(b)]. Furthermore, when 1 day (24 h) had passed from the treatment start [Fig. 7(c)], it was observed that the resin portion had swelled and that the decomposition of the cured resin had advanced the most. Two days (50 h) from the treatment start [Fig. 7(d)], the metal substrates could be dissociated from the resin portion and recovered. When the decomposed resin portion in this system was observed, the resin portion was decomposed very finely compared with the unmodified resin or the modified resin with homogeneous morphology. Thus, the modified resin with continuous-phase morphology of PES recovered the molded metal substrates in a shorter time compared with the other cured resins.

The relation between the number of treatment dates of these cured resins and the pulling-out adhesion strength is shown in Figure 8. The strength before treatment (0 days) shows the pulling-out adhesion strength of these cured resins. The pulling-out adhesion strength of the unmodified resin was about 30 MPa. The adhesion strength of the modified resin with homogeneous morphology was 28 MPa, and the value

was almost equal to the unmodified resin. In the cured resin with continuous-phase morphology of PES, the adhesion strength improved to about 34 MPa, and was about 1.1 times greater than that of the unmodified resin. It was indicated that the adhesion strength improves by addition of PES to the epoxy resin. When solvent treatment of the piece of adhesion examination of these cured resins was carried out, the strength decreased with the progress of the number of treatment dates. However, the decreased speed of the strength in each cured resin differed greatly. In the modified resin with continuous-phase morphology of PES, the strength decreased rapidly immediately after the treatment start. In addition, the number of treatment dates to which the adhesion strength results in 0 MPa was 2 days for this system. On the other hand, in the unmodified resin and the cured resin with homogeneous morphology, strength on the second day from the treatment start was 20 MPa or more, and high strength was maintained. In the unmodified resin and the modified resin with homogeneous morphology, the strength decreased gradually from the start of the solvent treatment. For the modified resin with homogeneous morphology, the number of treatment dates to which the adhesion strength results in 0 MPa was 13 days. The adhesion strength of the unmodified resin at this time was still about 7 MPa, and it was 25 days after the treatment start that the strength of the unmodified resin was 0 MPa.

As mentioned above, when the thermoplastic polymer was blended with the thermosetting resin, and the dispersed state of the thermoplastic polymer was controlled so that the thermoplastic polymer formed a continuous phase, the thermosetting resin decomposed easily with chemical (solvent) treatment at the time of abandonment.

Furthermore, the material that had a continuous variation in the phase morphology of a cured resin from the portion with homogeneous morphology to the portion with continuous-phase morphology of PES, that is, continuously variable morphology material, was created by attaching a slope to the molding temperature of a sample, as shown in Figure 9. The decomposition speed in chemical treatment is controllable by adding the variation in the phase morphology. If the portion to be disassembled is controlled to the continuous phase of the thermoplastic polymer, decomposition of a product and recovery of molded materials can be easily performed by the chemical treatment, etc.

Effect of the morphologies of the cured resins modified with PES on the thermal-mechanical properties

The thermal and mechanical properties of the cured resins modified with 20 wt % of PES were evaluated.

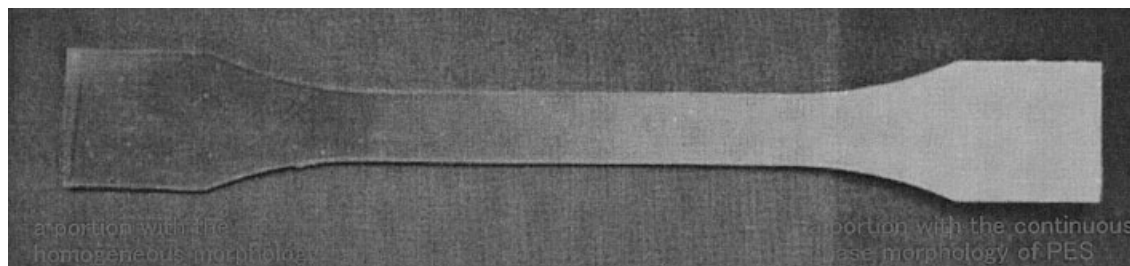


Figure 9 Material with a continuous variation in the phase morphology of the cured resin. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

The results are shown in Table I. The T_g of the unmodified resin showed 150°C. The T_g of the cured resin formed the continuous-phase morphology of PES was 160°C and shifted about 10°C higher than that of the unmodified resin. In the cured epoxy resin with homogeneous morphology, the value of the T_g showed about 180°C and rose by about 30°C compared with the unmodified resin. Thus, when PES was added to the epoxy resin, the heat resistance of the epoxy resins improved by 10–30°C. It can be concluded that significant improvement in the T_g of the cured resin with homogeneous morphology is attributed to the formation of the semi-interpenetrating polymer networks (semi-IPN) structure composed of the epoxy network and linear PES as our previous report described.¹⁵

The values of the flexural strength and flexural modulus of these modified resins were equal to that of the unmodified resin independent of their morphologies. However, the elongation at the break of these modified resins was dependent on them. The elongation of the modified resin with the continuous-phase morphology of PES was equal to that of the unmodified resin. On the other hand, the value of the elongation of the modified resin with the homogeneous morphology was about 1.4 times greater than that of the unmodified resin. This is attributed to the effect of the formation of the semi-IPN structure. The fracture toughness of these modified resins was also dependent on their morphologies. In the resin with the homogeneous morphology, the fracture toughness increased with the addition of PES, and its value was about 1.5 times greater than that of the unmodified resin. The fracture toughness of the modified resin

with the continuous-phase morphology of PES significantly increased, and its value was about 1.9 times greater than that of the unmodified resin. Although our previous report¹⁵ described the fracture mechanism in detail, a significant increase in the fracture toughness was achieved when the PES with high toughness formed the continuous phase in the cured resin. Thus, the addition of PES to the epoxy resin led to improvement in the heat resistance and toughness of the cured resin.

Figure 10 shows the temperature dependencies of the viscosity of the epoxy resins modified with 20 wt % PES. As for the resin viscosity, the viscosities of both the unmodified resin and the modified resin with 20 wt % of PES decreased with an increase in temperature. However, the viscosity of the resin modified with 20 wt % of PES was significantly increased compared with the unmodified resin. The value was about 1500 times that of the unmodified resin.

As mentioned above, although the addition of PES to the epoxy resin improves the heat resistance and toughness of the cured resin, it leads to a significant increase in the viscosity of the resin and results in a decrease in handling. In the future, technical development that depresses the increase of viscosity of the resin will be important.

CONCLUSION

To recover/reuse valuable metal materials from waste at the time of abandonment, development of an easy decomposition thermosetting resin was attempted. This resin was created by blending PES of a thermo-

TABLE I
Mechanical Properties of PES-Modified Resins

| Sample | Flexural properties | | | K_c (MN/m ^{3/2}) | T_g (DMA) (°C) |
|----------------------------|---------------------|---------------|------------------|---------------------------------|---------------------|
| | Strength (MPa) | Strain (%) | Modulus (GPa) | | |
| Unmodified resin | 123 ± 6 | 6.50 ± 1.54 | 2.71 ± 0.09 | 0.83 ± 0.03 | 151 |
| PES-modified resin (140°C) | 121 ± 2 | 9.20 ± 2.57 | 2.67 ± 0.03 | 1.22 ± 0.01 | 179 |
| PES-modified resin (180°C) | 127 ± 7 | 7.14 ± 2.83 | 2.89 ± 0.01 | 1.55 ± 0.08 | 159 |

PES content : 20 wt %.

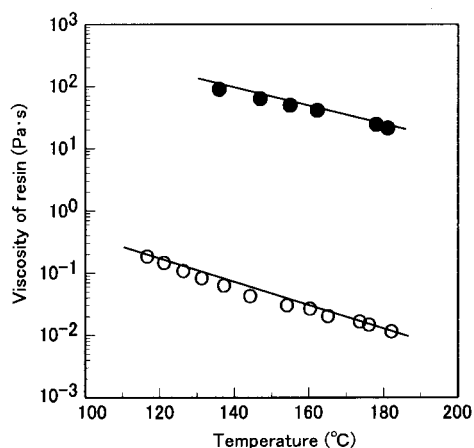


Figure 10 Effect of the addition of PES on the viscosity of the epoxy resins before curing. Sample: ○, unmodified resin; ●, epoxy resin containing 20 wt % of PES.

plastic polymer that is soluble in a solvent or heat with the epoxy resin of the thermosetting resin, which is insoluble. The dispersion state of the PES in the epoxy resin was controlled by changing the molding temperature. The influence of the morphology of the cured resin upon the decomposition nature by external stimulus of the organic solvent was investigated. The following results were obtained.

(1) In the unmodified resin and the modified resin with homogeneous morphology, the cured resins did not decompose easily when they were dipped in the organic solvent DMF at 23°C. It took 13 days or more to recover the metal materials molded into the cured resin. On the other hand, in the modified resin with continuous-phase morphology of PES, decomposition proceeded rapidly when it was dipped in DMF (23°C). The resin portion was decomposed completely in only 2 days, and the molded metal material was recoverable.

(2) The heat resistance (T_g) of the cured resins modified with PES improved by 10–30°C compared with the unmodified resin. Moreover, the fracture toughness value also improved about 1.5–1.9 times more than that of the unmodified resin.

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