Effect of Electrical Field on Crazing Test of Enameled Wire in Water

SHIGEO MASUDA and NOBUYUKI ASANO, *Sumitomo Electric Industries Co., Nagoya, Japan*

Synopsis

We investigated the tendency to crazing to enameled wire under applying DC voltage in an electrolyte in the different conductivity. Crazing occurred in the enameled wire film in the case of connecting the wire to the negative pole, but did not occur in the film in the case of connecting the wire to the positive pole. It was found that the penetration of H_3O^+ ion, due to electroosmosis, is the major factor for crazing.

INTRODUCTION

Enameled wire is manufactured by coating thermosetting resin on a conductor and baking it in an oven, alternately repeating these processes. Results of our experiments on the crazing that occurs in enameled wire have been reported in papers published by the Japanese Society of Rheology. In these papers, we clarified the following:

1. Crazing in enameled wire is most severe at 3–5% elongation, where the residual stress of the film is maximized. Thus, crazing occurs to relieve this residual stress.[1]

2. The occurrence of crazing in enameled wire varies greatly with the type of solvent used. Any type of solvent, it coated after elongation, acts on enameled wire in one of two ways: the film shrinks, causing the residual stress to temporarily increase, or the residual stress of the film is relaxed.[2]

3. A solvent that tends to cause crazing also has a tendency to cause a decrease in the glass transition temperature of the film after the enameled wire is immersed in the solvent.[2]

4. Crazing of a certain kind of enameled wire can be eliminated through heat fusion if the crazed wire is heat-treated at a temperature above the glass transition temperature of the film.[3]

There is an evaluation method currently in wide use for crazing characteristics of enameled wire. This method applies DC voltage to a wire immersed in electrolyte, with the wire as negative and the liquid as positive in polarity. In such a test, the film tends to become crazed on enameled wires susceptible to crazing and, at the same time, it can be observed that gas is generated from the surface of the film by the electrolysis of water. This method, based on the enameled wire pinhole test, is widely used for testing the crazing of enameled wire in water.[4]

We conducted a number of studies on phenomena occurring during such crazing tests and now report the results as follows.

Journal of Applied Polymer Sciences, Vol. 29, 1309–1318 (1984)
© 1984 John Wiley & Sons, Inc.
CCC 0021-8995/84/041309-10$04.00

TABLE I
Enameled Wires[a]

Enameled Wire	Enamel	Film thickness (mm)	Tg (°c)
PVF	Polyvinyl formal	0.040	110
PEW	Polyester	0.040	128
UEW	Polyurethane	0.040	135
EIW	Polyesterimide	0.039	195
AIW	Polyamideimide	0.040	285

[a] Conductor diameter 1.0 mm.

EXPERIMENTAL

Test Samples. Five kinds of enameled wires for use as coils in motors and transformers were employed as test samples (Table I). These test samples were prepared by oxidizing a copper wire, coating an insulating meterial on it, and baking it in an oven. Thus, adhesion of the film to the copper wire was reduced to allow assessment of the change in stress only in the film when crazing was caused to occur by the method in the subsection below. NaCl solution of various concentrations was used as the electrolyte (Table II).

Change of Residual Stress in Coated Film Caused by Crazing. Each test sample was first held in place by the chuck of a tension testing device (Autograph S-100 made by Shimadzu Corp.). Then gauze was wound in three layers around the test sample, with a 0.3-mm-diameter bare copper wire loosely wound around for use as an electrode. For electrode connection to a DC current, the test sample was used as the negative pole, and the bare copper wire as positive in polarity. Gauze functioned to retain the electrolyte applied to the surface of the test sample (Fig. 1).

Figure 2 shows the change of residual stress in the film caused by crazing that occurred in the PVF-enameled wire used as a test sample. With the test sample held in place, elongation began at time t_0, where stress increased (Fig. 2, OA). When the elongation stopped at time t_1, the stress was relaxed from A to B. NaCl solution was dripped onto the gauze by using a filler at time t_2 to wet the test sample surface. Then DC voltage was applied at time t_3. This caused crazing to occur, resulting in relaxed residual stress in the film, from C to D. When stress–relaxation as a result of crazing became almost stable at t_4, a tangent was drawn from D to curve CD. With the intersection of extrapolated line CG designated as H, CH represents the amount of stress relaxation resulting from the crazing. If a cut is made with a knife around the circumference of the film at t_4, residual stress DE in the film due to crazing can be determined. During the test, the elongation rate of the test sample was set at 5%.

TABLE II
NaCl Aqueous Solution

Concn (%)	0.0	0.2	0.4	0.6	0.8	1.0
Specific resistance (Ω · cm)	850×10^2	16×10^2	2.0×10^2	1.7×10^2	1.4×10^2	1.1×10^2

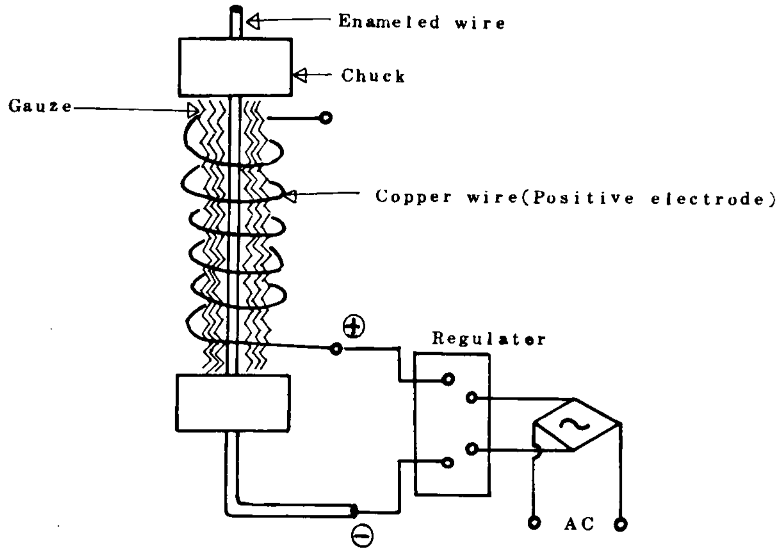

Fig. 1. Test method for evaluating the effect of DC voltage on the crazing of enameled wire.

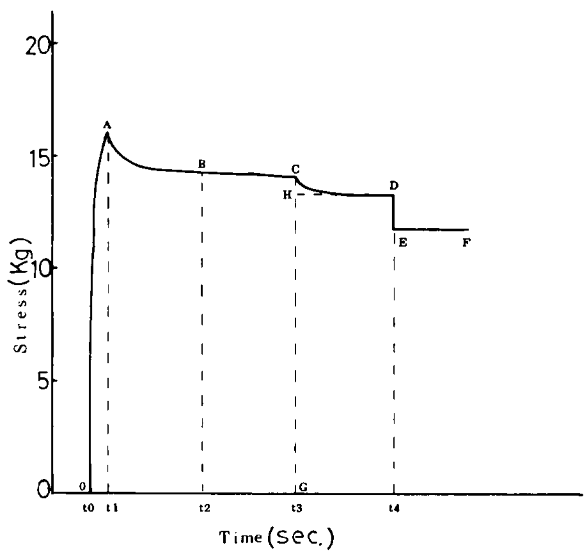

Fig. 2. Stress–relaxation of enameled wire film due to crazing produced by applying DC voltage. Enameled wire, PVF; conductor diam, 1 mm; $t_1 - t_0 = 10$ s; $t_2 - t_1 = t_3 - t_2 = t_4 - t_3 = 60$ sec.

TEST RESULTS AND DISCUSSION

Crazing and Stress–Relaxation Resulting from DC Voltage Applying. Using 0.2% NaCl solution, a comparison was made to check the occurrence of crazing in the test samples (Table III). As is clear from Table 3, both PVF and EIW enameled wires tended to become crazed. No crazing occurred in PEW, UEW, and AIW even if DC 150 V was applied for 60 min. The enameled wires

TABLE III
Enameled Wire vs. Condition for Crazing Occurrence

Enameled wire	Condition for crazing occurrence
PVF	Occurrence within 1 min by applying 1 V
PEW	No occurrence within 60 min by applying 150 V
UEW	No occurrence within 60 min by applying 150 V
EIW	Occurrence within 1 min by applying 20 V
AIW	No occurrence within 60 min by applying 150 V

that are resistant to crazing will be further studied and results will be reported in detail. This report presents test results mainly on PVF and EIW.

In addition to the test method under discussion, there is another crazing test method in which a test sample is immersed in a solvent after it is elongated. Although different in the extent of crazing when a polar solvent with a high dielectric coefficient was used, this method appeared to cause crazing on all enameled wires tested for this report. For this reason, the crazing test method using water is considered milder than that using a solvent.

Concentrations of NaCl Solution and Applied Voltage. Figure 3 shows the relaxation of stress as a result of crazing when the voltage level applied to the PVF enameled wire was varied. Figure 4 shows the amount of stress relaxation by the time t_4 in Figure 3 after both the applied voltage level and concentration of NaCl solution were varied. It was evident that the higher the applied voltage of the higher the concentration of NaCl solution, the more stress was relaxed as a result of crazing.

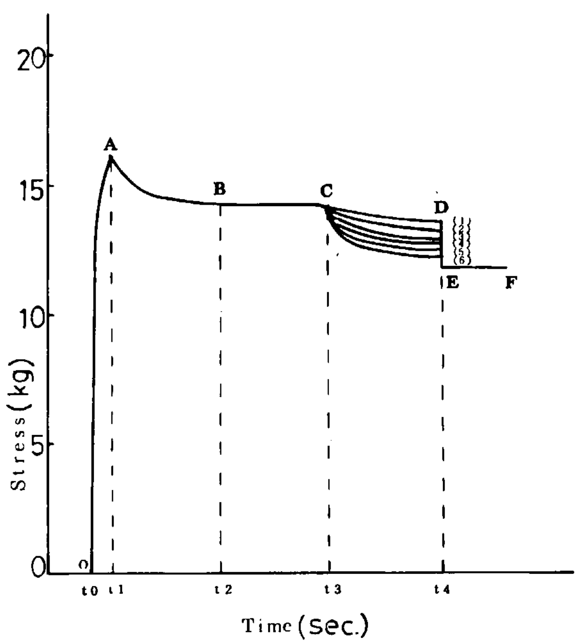

Fig. 3. Stress-relaxation of PVF film due to crazing produced by applying DC voltage. $t_1 - t_0 = 10$ s; $t_2 - t_1 = t_3 - t_2 = t_4 - t_3 = 60$ s. DC voltage V: (1) 1; (2) 3; (3) 6; (4) 9; (5) 12; (6) 15.

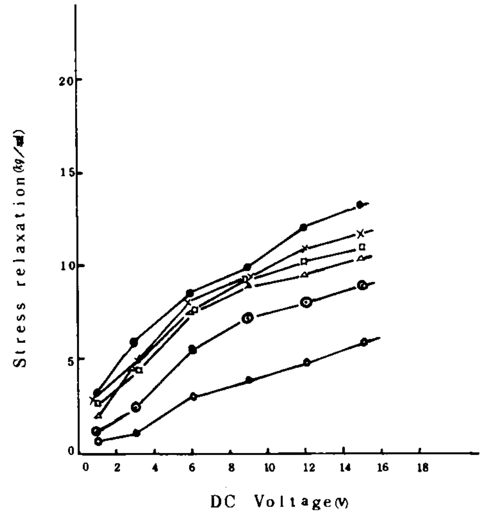

Fig. 4. Dependence of stress–relaxation due to crazing on voltage and concentration of NaCl solution (%): (○) 0.0; (⊙) 0.2; (△) 0.4; (□) 0.6; (X) 0.8; (●) 1.0.

Figure 5 is an optical microphotograph showing crazing that occurred when DC 3 V was applied to a 5%-elongated PVF enameled wire for 1 min. Figure 6 is an optical microphotograph showing the crazing that occurred when 12 V was applied for one minute. As is clear from the photographs, the higher the voltage, the wider the width of crazing caused. This means that stress–relaxation caused by crazing increases with the level of voltage applied.

Degree of Baking of Enameled Wire Film and Occurrence of Crazing. Using EIW enameled wire, experiments were also conducted to check the relation between the degree of baking of the film and the resistance to crazing (Table IV). The occurrence of crazing depended on the degree to which the film was baked, becoming less frequent as the amount of baking was increased and the glass transition temperature rose. It was also evident that a higher voltage applied over a longer period was necessary to bring about crazing on intensely baked film. Compared with that on the PVF-enameled wire film, the occurrence of crazing on the EIW-enameled wire film was significantly less. However, an

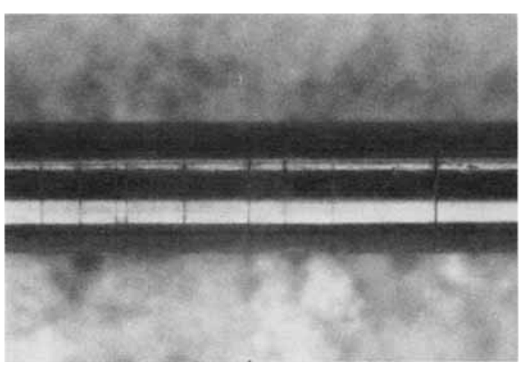

Fig. 5. Crazing of PVF produced by applying DC 3 V for 1 min.

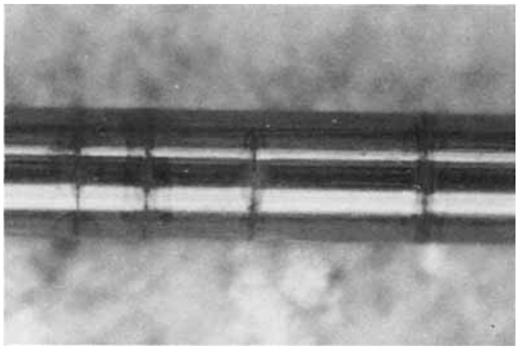

Fig. 6. Crazing of PVF produced by applying DC 12 V for 1 min.

unusual phenomenon was observed during the test: the residual stress of the film was relaxed by less crazing, with crazing occurring over the entire circumference of the film. As a result, the film was completely cut off.

Relationship between Polarity of Applied Voltage and Occurrence of Crazing. In the crazing test performed in water, the enameled wire was connected to the negative pole, and the electrolyte was used for positive polarity. We did an experiment with these polarities reversed, and observed interesting phenomena. While the PVF enameled wire was connected to the positive pole, no crazing occurred, as shown in Figure 7. There was thus no relaxation of stress on the film that otherwise would have occurred by crazing. However, as soon as the polarity was reversed, crazing occurred on the film and, as a result, the residual stress of the film was relaxed significantly.

Judging from this, it was considered probable that some substance could have attacked the film in the presence of an electrical field while the crazing test was

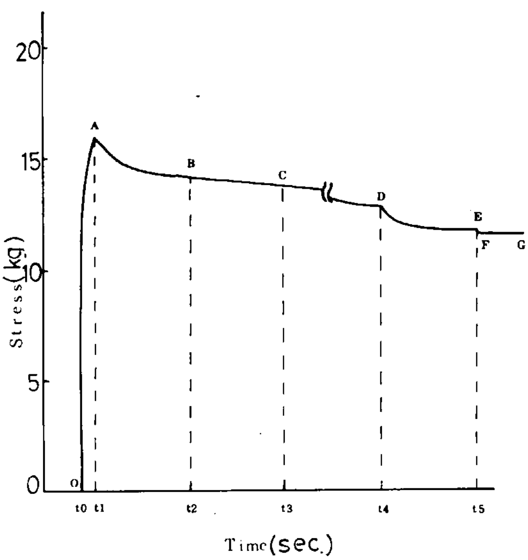

Fig. 7. Stress–relaxation of PVF film due to crazing: (t_2) applying NaCl aq. solution; (t_3) applying DC voltage connecting the wire to the positive pole; (t_4) applying DC voltage connecting the wire to the negative pole; $t_1 - t_0 = 10$ s; $t_2 - t_1 = 60$ s; $t_3 - t_2 = 60$; $t_4 - t_3 = 600$ s; $t_5 - t_4 = 60$ s.

TABLE IV
Undercured, Optimum Cured and Overcured EIW vs. Condition for Crazing Occurrence

	Applying voltage (DC, V)									
	1	3	6	9	12	15	20	30	40	50
Undercured EIW (T_g = 160°C)	3 min	1 min	38 s	18 s	12 s	7 s	3 s	—	—	—
Optimum cured EIW (T_g = 188°)	>30 min	>30 min	>30 min	>30 min	22 min	10 min	1 min	30 s	14 s	5 s
Overcured EIW (T_g = 210°C)	>60 min	>60 min	>60 min	>60 min	>60 min	>60 min	>60 min	50 min	10 min	1 min

performed in water. Therefore, we turned our attention to the electroosmosis phenomenon,[5] which is one of the electric field phenomena. Since then, overall studies were developed to explore the relation between the electroosmosis phenomenon and crazing.

Electroosmosis and Crazing Occurrence Mechanism. The electrode reactions occurring both in the positive and negative polarities during the electrolysis of water can be represented by formulas (1) and (2) below.

$$\text{Negative} \quad H_3O^+ + e \rightarrow H_2O + \tfrac{1}{2}H_2 \qquad (1)$$

$$\text{Positive} \quad OH^- \rightarrow \tfrac{1}{2}H_2O + \tfrac{1}{4}O_2 + e \qquad (2)$$

During the tests, voltage was applied to the elongated test sample, which was used as the negative pole. Microscopic observation of the test sample surface during the test disclosed that crazing occurred gradually and that hydrogen gas was generated from the crazed portions. On the other hand, when the elongated test sample was connected to the positive pole, neither crazing nor gas generation occurred. Therefore, we concluded that crazing only occurred when the test sample was connected to the negative pole. This is probably because the intermolecule force of the film chain becomes extremely weak when H_3O^+ ions are transpositioned into the film due to electroosmosis. As a result, the intermolecule force is unable to bear the residual stress of the film, and thus crazing occurs.

Electroosmosis is a phenomenon which occurs, for example, when a electrolyte is divided into chambers by porous ceramic plates, each of which is provided with positive and negative polarities. When DC voltage is applied to them, the positive electrolyte generally flows into the negative chamber.

NaCl solution of various concentrations used for our experiments was divided by ceramic cylinders, each containing a certain concentration, and the amount of solution flow due to electroosmosis was measured. In this case an electrode in the ceramic cylinder was used as the negative pole, using the applied voltage as a variable. The test results disclosed that the higher the concentration of NaCl solution and the applied voltage, the greater the amount of solution moved by electroosmosis. This conforms to the fact that the amount of stress–relaxation caused by crazing increases as both the concentration of NaCl solution and applied voltage increase.

Next, to directly confirm whether the electrolyte could permeate the enameled wire film by electroosmosis, we checked if there was any change in the weight of nonelongated enameled wires respectively connected to negative and positive polarities, using the concentration of NaCl solution and the applied voltage as variables. As a result, virtually no change was observed in the weight of the wire connected to the positive pole. However, an increase was obvious in the weight of the other wire, connected to the negative pole (Table V). The higher the concentration of NaCl solution and applied voltage, the greater the rate of weight increase. According to these test results, it was concluded that the occurrence of crazing during tests in water was obviously caused by the electroosmosis of H_3O^+ ions into the film. However, it was also concluded that, during crazing tests in water, even the slightest crazing may be prevented by expansion of the film due to penetration of the H_3O^+ ion component through the effect of electroosmosis, and by the resultant stress–relaxation. This concept is identical

TABLE V
The Change of Weight of PVF-Enameled Wire (Δm)[a]

Applying Voltage (V)	Concn (%) of NaCl aq. solution 0.0	0.2	0.4	0.6	0.8	1.0
1	0.1 mg	0.1 mg	0.2 mg	0.2 mg	0.3 mg	0.5 mg
3	0.2	0.3	0.6	0.9	1.0	1.2
6	0.2	0.4	0.7	1.0	1.4	1.7
9	0.4	0.7	0.9	1.4	1.7	2.2
12	0.6	1.3	1.7	2.1	2.6	2.9
15	0.9	1.7	2.2	2.6	2.9	3.4

[a] Length of enameled wire was 1 m. $\Delta m = m - m_0$, where Δm = the change of weight of enameled wire, m = the weight of enameled wire after applying voltage, m_0 = the weight of enameled wire before applying voltage. DC voltage was applied for 30 min.

TABLE VI
Conductive Liquid vs. Stress–Relaxation Due to Crazing and the Change of Weight Due to Electroosmosis[a]

Conductive liquid	Concn (%)	Specific resistance ($\Omega \cdot$ cm)	Stress–relaxation due to crazing (kg/mm^2)	Change of weight of enameled wire (mg)
HCl	0.2	60×10^2	2–3	0.4
H_2SO_4	0.2	76×10^2	2–3	0.4
HNO_3	0.2	65×10^2	2–3	0.5
NaOH	0.2	2×10^2	6–7	1.7

[a] DC 15 V was applied for 30 min. Length of PVF-enameled wire was 1 m.

to that in which a solvent permeates the film, which is expanded to promote stress–relaxation, thus preventing the occurrence of crazing.

Actually, crazing takes place during crazing tests using either water or solvent. Although the film can be expanded under the action of H_3O^+ ions, the relaxation of stress may still be insufficient to completely prevent the occurrence of crazing.

Electrolyte. We also used a variety of acid and alkaline salt solutions to study the stress–relaxation caused by crazing and also electroosmotic characteristics. Table VI shows the stress–relaxation caused by crazing and the change in weight due to electroosmosis. As is clear from Table VI, alkaline salt solutions are more likely to cause crazing than are acid solutions. Generally, the film formed over the enameled wire proved to be more vulnerable to alkali than to acid, as observed through a variety of chemical tests on enameled wire. The cause of this tendency remains to be explored as an important topic of investigation.

CONCLUSIONS

Crazing occurred in the test of enameled wire in water, in which the samples were used as the negative pole and the NaCl solution as the positive polarity. This crazing occurs as a result of electroosmosis of H_3O^+ ions in the film of the wire to cause the intermolecule force of the film molecule chain to decrease. The chain is then no longer able to withstand the residual stress, and crazing occurs.

These characteristics were verified in that the film weight increased only when the test sample was connected to the negative pole, whereas it remained unchanged when the test sample was connected to the positive pole.

Our experiments proved that the higher the concentration of NaCl solution and the applied voltage, the easier crazing occurs. It was therefore concluded that the transposition of H_3O^+ ions into the film by electroosmosis was accelerated as both the concentration of NaCl solution and the applied voltage increased.

References

1. S. Masuda and N. Asano, *J. Appl. Polym. Sci.*, **20**(2), 357 (1976).
2. S. Masuda and N. Asano, *J. Soc. Rheol. Jpn*, **4**(4), 154 (1976).
3. S. Masuda and N. Asano, *J. Soc. Rheol. Jpn.*, **5**(3), 117 (1977).
4. "Methods of Test for the Enameled Copper Wire and Enameled Aluminium Wires: Pin-Hole," JIS C 3003, Japanese Standard Association.
5. T. Takahashi, *General Description of Electrochemistry*, Maki Shoten, Japan, 92.

Received June 28, 1983
Accepted September 15, 1983