

## Study on the Causes of Premature Cracking of Epoxy Coatings for Ship's Ballast Tanks

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Premature cracking of the epoxy coatings applied on ship's ballast tanks(BT) can lead to damage of ship's hulls. To avoid this, it's important to have clear understanding of the underlying mechanism and primary factors of the coating crack. In this study, the efforts were made to clarify the integrated effects of main factors, i.e., initial coating shrinkage, thermally induced strain, steel-structural strain and the intrinsic coating flexibility at the initial and after aging, to the early cracking phenomena of epoxy coating in the ship's ballast tank.

The coating crack is caused by combination of thermal stress, structural stress, and internal stresses which is closely related to chemical structures of the coatings. On the other hand, thermal stresses and dimensional stabilities would rarely play a major role in coating crack for ballast tank coatings with rather large flexibility. Crack resistance of the coatings at early stages can be estimated roughly by measuring internal stress, FT-IR and  $T_g$  value of the coatings. A new screening test method was also proposed in this study, which can be possibly related to the long-term resistance of epoxy-based paints to cracking.

**Keywords** : coating crack, ballast tank protective coating, flexibility, internal stress, thermal stress, structural stress

### 1. Introduction

Recently, several Crude Oil Tankers(COT) were reported to experience early failure of protective coatings in the form of brittle cracking. Cracking of painted film notably in the Ballast Tank(BT) section is considered to be a potential risk to the structure integrity of ship. While coating maintenance and corrosion control of ship ballast tanks is costly and difficult job, surprisingly little is reported about the cause and remedy of this type of coating failure. Generally, the early cracking of painted films on ship's hull is attributed to the internal/external stresses in the film generated by combination of four main factors as follow:<sup>1)</sup>

- Shrinkage due to chemical curing
- Migration and loss of low-molecular components after initial shrinkage
- Strain of the steel substrate
- Environmental impacts (temperature change, etc.)

However, regarding the dominant factor controlling the coating crack there are quite a few disagreements among the several studies reported by different groups. For exam-

ple, O. Negele et al. demonstrated that internal stress by curing shrinkage in organic coatings was the main cause of cracking and checking,<sup>2)</sup> whereas B. J. Zhang et al. argued that coating cracking is caused not by the internal stress of cure shrinkage, but by thermal stress during water ballasting/deballasting.<sup>4)</sup>

Based on the survey reports of COT's BT sections, cracking of epoxy coating was reported to be concentrated on highly stressed areas such as fillet welds, transitions between structural details, weld toes, etc. Local stresses on these areas may reach or even exceed the yield strength of steel. Recent trend in ship building is to employ high strength steel plates with less thickness, which results in greater flexing during operation of the vessel. As the result, it can be easily expected that unexpected cracking is more severe for epoxy coating applied on the high strength steels than for those on normal strength steels.<sup>5)</sup>

These findings indicate that the local structural stress on the epoxy coatings, incurred by steel substrate, will bear a direct effect on the coating cracking on those areas. Nonetheless only a few research works have given attention to this type of local structural stress aspect of coating crack than to the other eligible factors. In this study, the efforts were made to clarify the integrated

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effects of above mentioned important factors, i.e., initial coating shrinkage, thermally induced strain, steel-structural strain and the intrinsic coating flexibility at the initial and after aging, to the early cracking phenomena of epoxy coating in the ship's ballast tank. A new screening test method was also proposed in this study, which can be possibly related to the long-term resistance of epoxy-based paints to cracking. In this approach, exposure time related epoxy chemistry and properties were evaluated, and used as a tool for determining the coating's long-term performance. For this purpose, the systematic relationship of simple evaluation criteria such as glass transition temperature, thermal expansion coefficient and Fourier Transform-Infrared Spectrometers (FT-IR) characteristics with coating crack resistance were tried to be obtained.

## 2. Preliminary study

Preliminary investigation has been performed to develop a reliable and simple method to achieve the object in this study. First of all, the amount of stress or strain on the local structural parts in ship's ballast tank was evaluated, especially, weld toes and heels which are known to experience the highest stress concentration, where the coating cracks were found more frequently. Structural design factors such as steel grade, maximum allowable stress and stress concentration factor were reviewed. For BT structure of COTs constructed with high strength steel, such as 315 MPa Yield strength(Y.S) grade steel, the maximum stress of 0.95 Y.S. is allowable on the concerned structural parts on the design base. However, the stress concentrated areas such as weld toes and heels would experience higher stress than other areas. In this respect, class regulations and codes provided the proper stress concentration factors for each type of connection of structural parts for design purpose.<sup>6)</sup> According to this regulation, the specific type of connection of structural parts for the concerned ship's WBT could be considered to suffer 2.25 of stress concentration factors at weld toes and heels. Therefore, the stress at weld toes and heels, actually, may reach to  $2.25 \times 0.95$  Y.S.. The corresponding strain applied on the epoxy coating on the steel parts was calculated to be 0.6% by Neuber's rule.<sup>7)</sup> The initial flexibility of the general epoxy coating is known to have an elongation in the range of 3-4%,<sup>1)</sup> whereas the polyamine adduct type epoxy resin may have lower value such as 1.5%.

Secondly, thermal data properties of epoxy coatings, as reported by other studies, showed that the coating's thermal expansion coefficient was estimated to be about  $100 \times 10^{-6}$  strain/ $^{\circ}\text{C}$  in the temperature range of 25~65 $^{\circ}\text{C}$ ,<sup>4)</sup>

whereas that of the steel is known to be  $12 \times 10^{-6}$  strain/ $^{\circ}\text{C}$ . These differences of thermal expansion between the coatings and steel substrate will cause a thermal stress in the coatings upon the presence of temperature gradient. Thus, the strain in the coatings induced by temperature increase from 20 $^{\circ}\text{C}$  to 60 $^{\circ}\text{C}$ , which COT's BT section encountered during the loading of heated cargo oil, may reach to the 0.35% strain in compression, and due to the nature of the stress, coating crack problem can be negligible in this stage. However, when the temperature is plunged from 60 $^{\circ}\text{C}$  to 20 $^{\circ}\text{C}$  or lower in a rapid cooling such as water quenching, which is expected in BT during cargo off-loading and water ballasting, the 0.35% strain may be fully reversed from the compression to tension mode. If the temperature of any baked polymer is reduced to pass through the glass transition ( $T_g$ ) range too quickly, it may lead to significant internal strain within the polymer film because an unfavorable molecular conformation may be frozen in place as the film temperature passes through the  $T_g$  range.

Finally, a result based on computer simulation analysis suggested that the internal stress of epoxy paint by curing shrinkage may be 8-10 MPa at a certain local area, corresponding to 33% of the tensile strength of epoxy paint film (24MPa),<sup>4)</sup> whereas the estimation from the ASTM manual indicated that the internal stress of the epoxy coatings cured at 20 $^{\circ}\text{C}$  and 50% RH will be about 5 MPa.<sup>8)</sup>

In this study, the effect of curing shrinkage of the epoxy coatings was elucidated by measuring the coating's maximum elongation at the break of the coated steel specimen. These elongation data was directly compared with the maximum allowable strain at the local parts of BT structure as well as with the thermally induced strain mentioned above. These comparisons were performed with the coating's elongation data at fresh state and after aging under simulated BT condition. Special environmental effects in ships' ballast tanks included cyclic elevated temperature on areas such as bulkheads adjacent to cargo holds with high humidity and water quenching condition.

## 3. Experimentals

Four different, commercial epoxy coatings, as designated by E, N, K, B, which are commonly used for COT's BT were investigated for both fresh and aged condition. Coating films with Dry Film Thickness(D.F.T.) of 300  $\mu\text{m}$ , 600  $\mu\text{m}$ , 900  $\mu\text{m}$  and 1200  $\mu\text{m}$  were made on Sa 2.5 blasted steel plate as well as in free film form, followed by ambient cure for fourteen days prior to evaluation. Each sample was evaluated for tensile strength, thermal stability, chemical structure, dimensional stability, glass transition

temperature, thermal expansion coefficient and internal stress.

**Tensile strength**

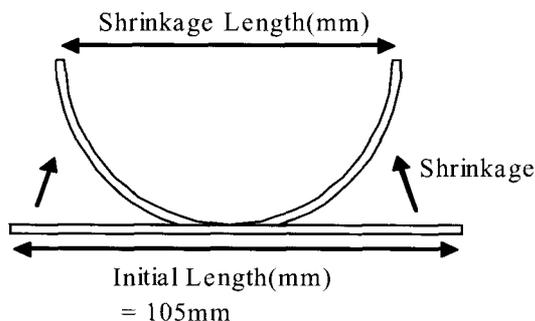
Tensile strength of each coating was measured being applied on Sa 2.5 dumbbell-model steel specimens by using a universal tensile machine with stain rate of 1 mm/min. and coating crack initiation was observed with a magnifier ( $\times 10$ ).

**Thermal stability**

The specimens for the thermal cycling test were prepared on steel T-bars ( $450 \times 120 \times 10$ mm). Each coating was applied with a gap of 150 mm distance so that the coatings of 300  $\mu\text{m}$ , 900  $\mu\text{m}$ , 1200  $\mu\text{m}$  were prepared on one specimen. The T-bar specimens were subjected to the following temperature cycle: 4 hours at 60°C, within 2 hours to 5°C, 4 hours at 5°C and within 2 hours back to 60°C. The relative humidity was 80%. Number of cycles was varied depending on the coating types and development of surface cracking, detachment and other defects, which were checked at a regular interval.

**Internal stress by the curing shrinkage**

Curing shrinkage of coatings was measured by the modified cantilever method.<sup>2)</sup> Coatings were applied at 300  $\mu\text{m}$  D.F.T. on Polyethylene Terephthalate (PET) film. The curing shrinkage ratio, then, was evaluated by the method, as shown in Fig. 1.



\*Shrinkage ratio(%) = (Initial length - Shrinkage length)/Initial length  $\times 100$

\*Shrinkage length is the diameter of the semi-circle shaped film

Fig. 1. Calculation method of shrinkage ratio

**Dimensional stability**

The dimensional stability of the coating was evaluated according to NACE standard TM0104-2004. Three free coating films ( $76 \times 25$ mm) were cured at room temperature for 2 weeks, then, initial D.F.T., length, width of each free-film test specimens were measured. After immersing

the test specimens in seawater at 60°C for 24 hours, their length and width were re-measured immediately.

**Aging**

Four different coating films were immersed in nature sea water at 20°C for 7days to simulate quenching effect on all films in their glassy state. These films were, then, heated at 60°C and 90% relative humidity for 7 days. This cycle was repeated up to 7 times.

**Thermal expansion coefficient, glass transition temperature, FT-IR**

Thermal expansion coefficient ( $T_a$ ) of the each coating was measured by increasing the temperature at 10°C/min. from 20°C to 120°C using Thermomechanical Analysis (TMA). To measure the glass transition temperature ( $T_g$ ) of the coatings, calorimetric data were obtained by using differential scanning calorimeter (DSC), and about 10 mg of a coating. Temperature range was 20°C to 120°C with a heating rate of 10°C/min. The glass transition temperature  $T_g$  was measured at the onset of the glass transition region. Fourier Transform-Infrared Spectrometers (FT-IR) was also used to analyze the molecular structure of 4 different epoxy resins and hardners. Attenuated Total Reflectance(ATR) spectra were collected from 32 scans at 4  $\text{cm}^{-1}$  resolution.

**4. Results and discussion**

**4.1 Elongation to break**

To evaluate the elongation of two coatings, E and B, the threshold strains for initiation of coating crack were measured using strain gauge. In the fresh condition, B paint exhibited better elongation than that of E paint, which was attributed to the difference in internal stress. It's generally accepted that the paint with higher curing

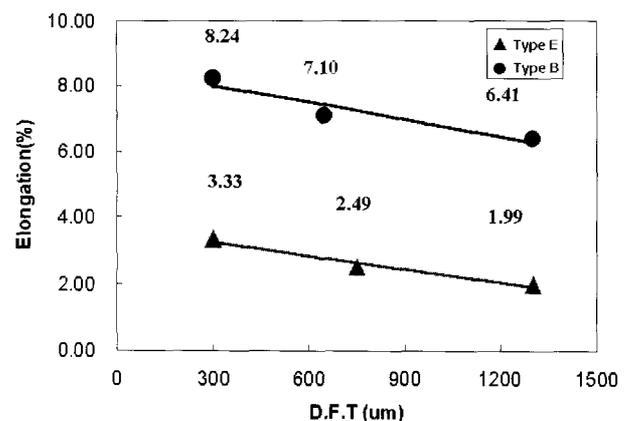


Fig. 2. Initial coating elongation in the fresh condition

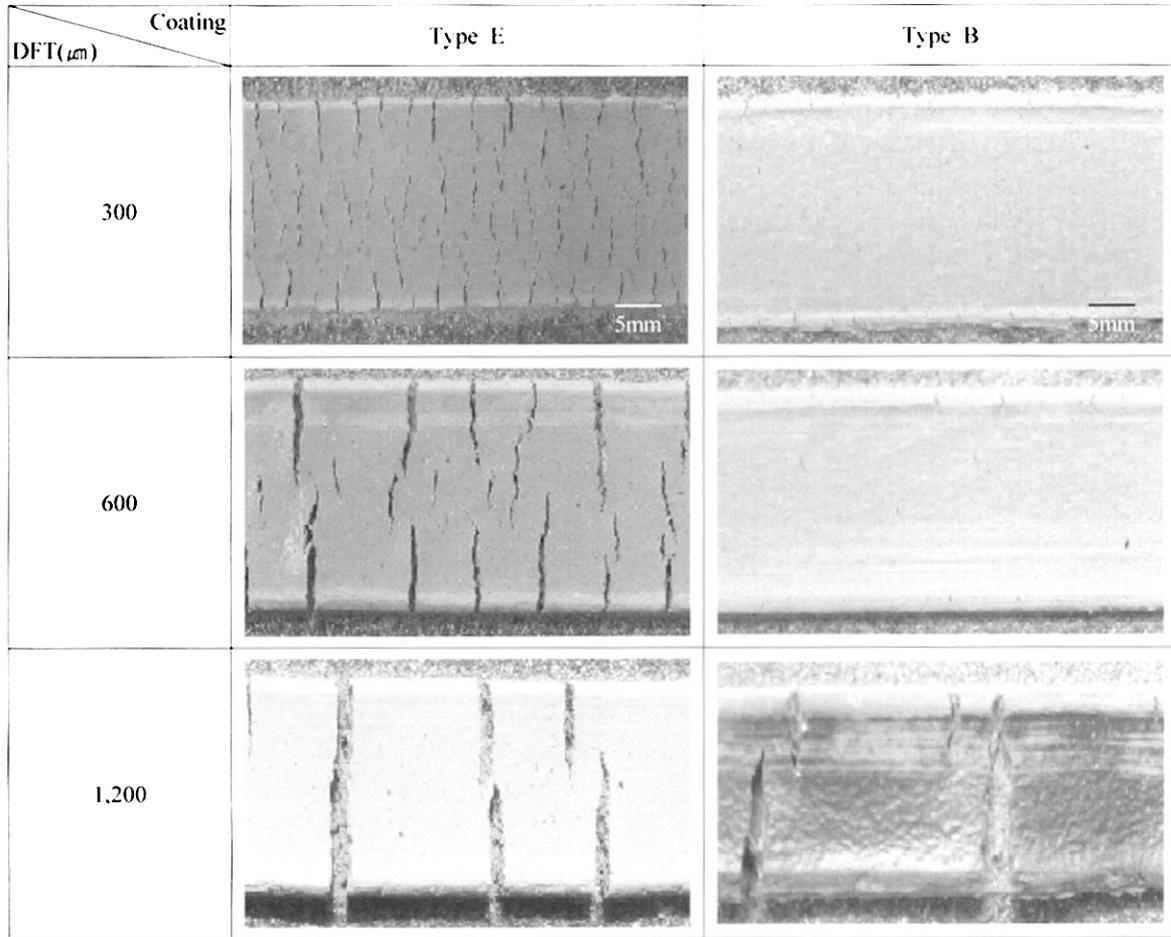


Fig. 3. Coating cracks developed after elongation test (elongated at 10%)

shrinkage would experience larger internal stress. During the coating film formation, its volume decreases due to curing and solvent evaporation.<sup>9)</sup> For the same reason, as coating thickness increases, its elongation property decreases linearly (Fig. 2). Increasing film thicknesses will increase internal stresses because of delayed solvent loss from the lower layers of the film, which explains that cracks in thicker films will be larger and more widely spaced than thinner films (Fig. 3).

#### 4.2 Evaluation of internal stress

Internal stresses originate during the film formation and curing processes as a result of solvent evaporation in all films and the cross-linking of thermosetting films. Both processes produce a volumetric compaction or shrinkage of the film. In adherent films, this shrinkage occurs with some resistance, which was from the adhesion of the film to the substrate. This resistance is a source of internal stress.<sup>10)</sup> The internal stress of paint film due to curing shrinkage, as shown in Fig. 4, would strongly affect its

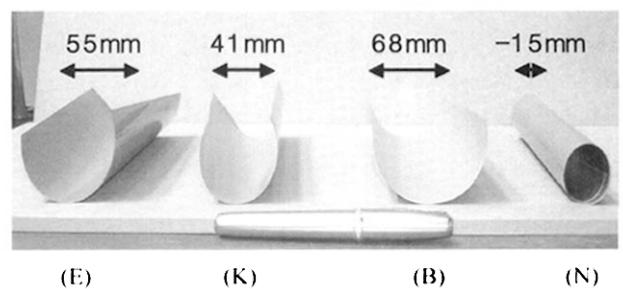


Fig. 4. Deflection by internal stress

cracking susceptibility, and it was reported that the initial curing shrinkage is one of the important factors for the paint film crack.<sup>2)</sup>

After four coating materials (D.F.T.=350 $\mu\text{m}$ ) were applied on to the PET film and fully cured, shrinkage length from the original flat form of each coating was measured as shown in Fig. 4. The deflections (curing shrinkage rate), of each coating calculated from Fig. 1, are shown in Table 1. The degree of curing shrinkage,

**Table 1. Degree of deflection (Shrinkage rate) for each coating**

Coating materials (SVR, %)	E (80)	K (68)	N (60)	B (60)
Length(mm)	55	41	-15	68
Shrinkage rate(%)	47.6	61.0	114.3	35.2

**Table 2. Thermal expansion coefficient and  $T_g$  for 4 coatings**

Coating materials	$T_g$ (°C)	$\alpha^1$ ( $\mu\text{m}/^\circ\text{C}$ )	$\alpha^2$ ( $\mu\text{m}/^\circ\text{C}$ )
E	54.65	98.0	95.0
K	54.64	130	164
N	49.93	145	217
B	48.82	181	223

$\alpha^1$ : Thermal expansion coefficient under  $T_g$ ,

$\alpha^2$ : Thermal expansion coefficient above  $T_g$

which is proportional to the internal stress, indicated that the internal stress of B paint is the lowest among those of the coatings. From the  $T_g$  data of Table 2, coating's internal stress would increase along the increase of its  $T_g$  value except in case of coating N. The  $T_g$  is affected by the structure of the final polymer and the reacting molecules. As the length of the polymeric segments between the functional groups decreases, restriction of molecular motion will be greater. In this case, the cross link density becomes higher, and the  $T_g$  will be higher.<sup>11)</sup> So the  $T_g$  plays an important role in dictating paint film behavior such as impact resistance, flexibility.

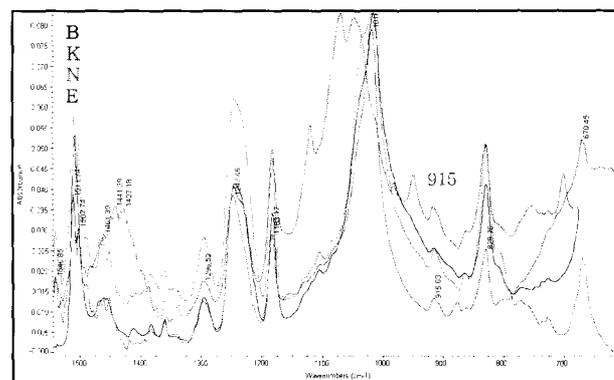
In the case of N coating, however,  $T_g$  value is lower (49.93°C) than others, but internal stress was measured to be much higher. The reason for this rather unexpected result can be explained as follows; because the amount of solvent in the N paint (Solid Volume Ratio, S.V.R., 60%) was much larger than those others, the solvent evaporation rate would be relatively slower. As solvent leaves the film, molecular "holes" or free volume spaces will be created into which the molecular segments of binder shall migrate.<sup>12)</sup> In thermosetting systems like epoxy, permanent primary bonds (cross linking) are formed between neighboring chains. These bondings and crosslinking in one segment of the polymer may severely restrict the ability of the adjacent polymer segments to migrate into the holes left as the vacant solvent molecules. This restriction is added up to a primary source of internal stress. Considering the previous report suggesting that internal stresses would only be occurred in the glassy region, increasing the stress of epoxies upon slow release of solvent from the film after initiation of the cross-linked structure formation.<sup>11),14)</sup> Thickness increase of these films would

cause stress (or strain) increase because solvent diffusion would continue after completion of solidification. This process seemed to be cause of great residual stresses observed in the some of coatings.

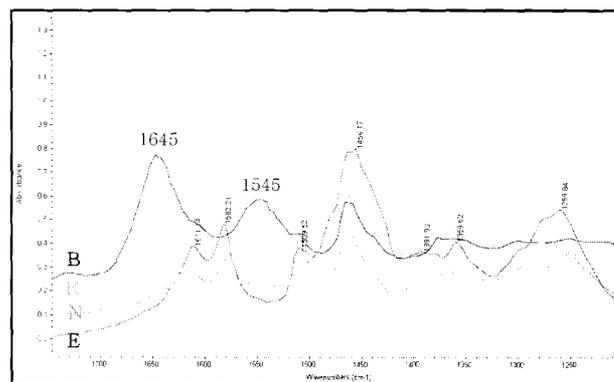
### 4.3 Evaluation of chemical structure

In the curing process of two-component epoxy paints, the epoxide group representing epoxy resin would react with various curing agents. The most commonly used curing agents for epoxy coatings are amines, amides, and phenol-formaldehydes. Upon completion of curing process, a three dimensional network of long chain molecules with limited flexibility will be formed, of which flexibility varies with the type and amount of curing agents.

Molecular weights of epoxy resins (such as, bisphenol A) can vary within the range of 350-1,000. Low molecular weight epoxies were originally considered unsuitable for protective coatings due to too short intermolecular distance between epoxide termination of the molecule, resulting in too high crosslinking density and low flexibility. Thus, it is quite important to look into molecular structure and characteristics of epoxy resin of each coating, to under



(a)



(b)

**Fig. 5.** FT-IR Spectra of 4 different epoxy coatings (a) resin, (b) hardener

stand their effects on the coating film's flexibility, and eventually their cracking susceptibility.

To evaluate chemical structure and molecular weight of coatings, ATR FT-IR measurements were carried out (Fig. 5). The result revealed that resins of E, N, B paints except K have the same chemical structure with different molecular weight. In the IR spectrum, absorption band observed at  $915\text{ cm}^{-1}$  indicated that the resin was epoxy, whereas higher peak suggested that its molecular weight is rather small, since the percentage of epoxy functional group in the molecule is relatively high. Molecular weight of B paint is the largest, while those of the others are similar. In the IR spectrum of B and K curing agent, peak at  $1645\text{ cm}^{-1}$  and near  $1545\text{ cm}^{-1}$  indicated the presence of amide linkage. Amide curing agents can decrease the crosslink density of the final epoxy, opening up the molecular network significantly. Even with relatively low molecular weight epoxies, this open molecular network gives coating films that are much more flexible and impact resistant than films cured with straight amines.<sup>15)</sup> These results were consistent with  $T_g$  data and internal stress data considering that molecular cross-link density would increase with lower molecular weight. By the same token, it can be said that a coating with higher  $T_g$ , higher shrinkage and larger internal stress will be incurred.

#### 4.4 Evaluation of thermal stress (hot/cold as per real running temperature)

In decreasing temperature environment coatings would be subjected to a tensile stress via volumetrically contraction, whereas, in increasing temperature, coatings would expand experiencing a compressive stress. The low temperature environment is particularly important as it cause and increase internal stress within the coating and therefore can be more destructive, which will lead to crack initiation and propagation. Reducing temperature causes the paint film to contract more than the steel. Because of the difference in the volumetric expansion coefficient between paint and the substrate, a shearing stress will occur at the interface.<sup>16)</sup>

Previous report on the same coatings used in this study showed that, E, N, B coatings did not show any crack or delamination during 30 thermal cycles (6 hours at  $-20^\circ\text{C}$ , within 6 hours to  $60^\circ\text{C}$ , 6 hours at  $60^\circ\text{C}$  and within 6hrs back to  $-20^\circ\text{C}$ ), even though max D.F.T. was  $2,000\text{ }\mu\text{m}$ . K paint and H paint, however, were reported to be failed on similar D.F.T. after 9 cycles and 12 cycles respectively in the previous study. So thermal cyclic test was carried on the coating K and the coating H. Coating E, N and B were excepted from the test because they didn't show any cracks in the previous study. Considering actual

BT service condition,  $-20^\circ\text{C}$  is far severer condition in terms of coatings' crack outbreak. Thus to simulate rather realistic thermal stress effect on the coating crack, in this study, experimental condition was set for the cyclic test between  $5^\circ\text{C}$  and  $60^\circ\text{C}$  representing actual BT service condition. As shown in Fig. 6, the welding joint area of the two coatings remained in good condition without crack after total 56 cycles. This difference in the test result, especially for K paint, which did not cracked in this study, is attributed to the effect of low temperature regime the cyclic thermal test.

The linear thermal expansion coefficient ( $\alpha$ ) of solid will vary markedly depending on the nature of the material. The higher the  $\alpha$  value, the larger expansion of the material would occur. The same goes for the contraction of the coating upon cooling stage. It was reported that the higher  $\alpha$  of coatings would result in more internal stress due to thermal stress. Prosser<sup>17)</sup> reported typical  $\alpha$  values for pigmented paint films were near  $4 \times 10^{-5}\text{ cm/cm}^\circ\text{C}$ , compared to  $1.1 \times 10^{-5}\text{ cm/cm}^\circ\text{C}$  for steel. Fig. 7 and Table 2 shows  $\alpha$  and glass  $T_g$  of different coatings, E, B, K, and N. As shown in Fig. 7, the  $\alpha$  values of paint films changed markedly with temperature increase through the

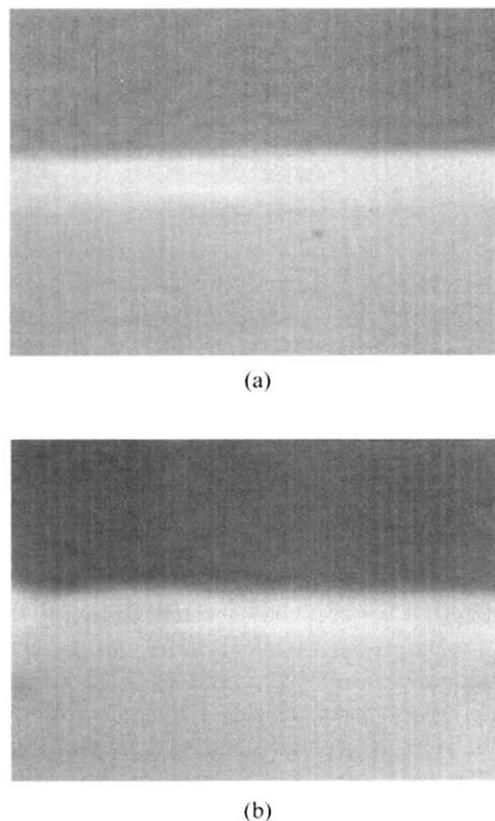


Fig. 6. Welding joint area after 56 thermal cyclic test (DFT  $1200\text{ }\mu\text{m}$ ); (a) coating K, (b) coating H

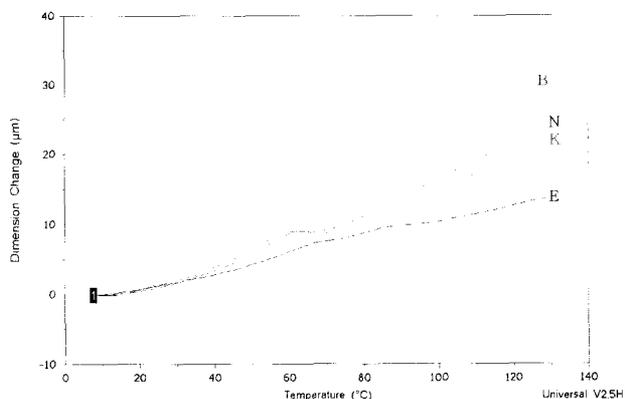


Fig. 7. Change of  $\alpha$  with exposure temperature for 4 coatings, measured by TMA

$T_g$  region. The strain of B and E paint at this test condition (from 60°C to 20°C) is 0.68% and 0.34% respectively because of the differences of thermal expansion coefficients. Volume expansion due to temperature change is known to be highly related to cross-link density of coatings, thus it can be interpreted as a good indicator of coating's flexibility. Therefore it can be easily assumed that the coating B might retain the best flexibility, whereas the coating E might reveal the worst flexibility after curing. In Fig. 7, it is quite noticeable that B paint, having the highest  $\alpha$  and high thermal stress, was far less prone to exhibit coating crack, which strongly suggested that thermal stresses would rarely play a major role in the development of coating crack contrary to the general assumption for some coatings with rather large flexibility.<sup>13)</sup>

#### 4.5 Evaluation of dimensional stability

For the BT coating, it was suggested that migration of moisture through the coating film (under wet and dry condition) is another factor for its cracking, since changes in the moisture content of coating films would create stresses, so called 'cyclic hygroscopic stresses'.<sup>18)</sup> Water absorption of the film in high humidity (or immersion) conditions and the subsequent desorption of water under dry conditions would cause dimensional distortions. The estimation from the ASTM code indicated that the hygroscopic strain of the coatings was proportioned to the R.H difference.<sup>3)</sup> Thus, the hygroscopic stresses are classified as a less dominant factor for coating cracking considering actual BT service condition, which was maintained high humidity in most times including deballasting period. During that period large amount of moisture would be condensed by heat of cargo tank. Dimensional changes of coating E was the largest, of which results were showed in Table 3. Volume expansion due to water absorption,

Table 3. Dimensional Change of the coatings

Coating materials	E	K	N	B
Before immersion (mm)	75.21 x 24.83 x 1.10	75.11 x 25.0 x 1.44	73.90 x 24.39 x 1.23	74.86 x 24.65 x 1.46
After immersion (mm)	75.21 x 25.08 x 1.15	75.21 x 24.80 x 1.46	74.34 x 24.37 x 1.22	74.62 x 25.21 x 1.46
% of expansion	1.86	0.24		0.65

contrary to temperature induced one, is closely related to chemical structure of resin and hardner, as well as cross-link density. For this very reason, this can not be directly used as an indicator of film's flexibility. From this data, coating E was expected to be aged more readily during service life.

#### 5. Summary

As a summary, several eligible cause of epoxy coating crack under ship's BT could be integrated as a function of aging time, as schematically visualized in Fig. 8. In this figure, it can be inferred that epoxy coating's elongation (or flexibility) is diminished by the amount of curing shrinkage at the initial state, and this will be cut down further by the strain induced by structural stress, thermal stress and hygroscopic stress. However, the initial coating's elongation of the most paints seems to be high enough to compensate these effects. On the other hand, aging of coating itself through thermal, wet/dry and mechanical fatigue condition would dwindle the coating's flexibility gradually with lapse of service time, which eventually resulted in the coating crack at some time.

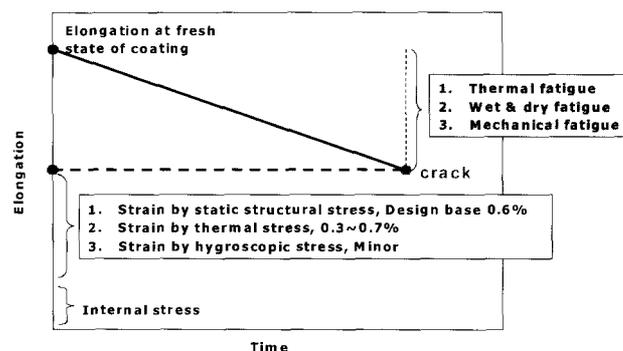


Fig. 8. Diagram for coating crack scenario

## 6. Conclusions

From several internal/external factors of coating for ship's BT, their impacts on coating cracking susceptibility were assessed. The coating crack is caused by combination of thermal stress, structural stress, and internal stresses which is closely related to chemical structures of the coatings. On the other hand, thermal stresses and dimensional stabilities would rarely play a major role in coating crack for BT coatings with rather large flexibility. Internal stresses of coatings can be measured by using a cantilever method and its chemical structure can be estimated by the FT-IR and  $T_g$  measurement. Thus crack resistance of the coatings at early stages can be estimated roughly by measuring internal stress, FT-IR and  $T_g$  value of the coatings. By employing this approach as a screening method, it was found that among the several commercial coatings, the coating B was found to retain an excellent potential for crack resistance. By using the same screening method, both K and N coating with higher  $T_g$  and larger internal stresses were expected to be cracked more easily. In terms of flexibility determination coatings under the aging condition, it was found that there was a wide difference among the coatings.

## 7. Future works

As for the next step to achieve the prevention of coating cracking in ship's BT, examination of the aging effect on crack resistance is necessary. For long term performance of these coatings, it is also necessary to look into how the characteristics of coating materials change with aging. Finally, the new test methods that can differentiate each coating's the long-term cracking resistance needs to be suggested, which will eventually lead to comprehensive measures to reduce or eliminate early cracking of WBT coatings in case of coating selection and coating application.

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