Study for Improvement of OIT accuracy for aging evaluation of organic material

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1. Introduction

Organic materials are generally aged by chemical reactions with oxygen. Antioxidant is one of additaments used to suppress the oxidative reaction of material. Volume of antioxidant decreases in proportion to time and degree of environmental condition such as temperature and radiation[1].

OIT(Oxidative Induction Times) measurement is a technique which can measure remaining volume of antioxidant in the material. Since oxydative induction time varies in proportion to amount of antioxidant remained after long term aging, it is possible to estimate the remaining life of the material by measuring OIT. Since OIT method requires approximately 5～10mg solid or powder specimen for test, nondestructive aging evaluation of organic material is possible[2].

Contrary to convenience of OIT method, reproducibility of OIT for same material was not so good due to reading problem of tangent line in OIT curve. Since the OIT curve had obtuse angle, accurate tangent line couldn't be made. It was found that inaccuracy of OIT came from the irregular shape of test specimen.

In this paper, process to find proper test specimen for best OIT accuracy is described.

2. Methods and Results

2.1 Introduction of OIT method

2.1.1 OIT theory

OIT is the time required to oxidize non-metal material when oxygen is introduced at constant temperature. Antioxidant is included in the non-metal material to protect oxidation of material. Heat can be generated because of reaction between oxygen and organic material when the antioxidant is exhausted due to long term aging. OIT is the time when the organic material become burning after complete loss of antioxidant. OIT has a relation with the volume of antioxidant remained in the material. Since there is abundant oxygen in the atmosphere, amount of antioxidant in the non-metal material decreases when the material is exposed at the atmosphere condition. Consequently, oxygen induction time become short. In this reason, OIT and aging condition have a proportional relation. It is also noted that OIT is being affected by temperature of surround. The higher environment temperature, the more reducing of antioxidant. This results in the shortening of OIT. We can measure the aging condition of material based on this character of OIT.

2.1.2 OIT measurement

DSC(differential scanning calorimeter) was used for the measurement of OIT. Thermal reaction is happened when oxygen is injected abruptly on test specimen in the DSC. 5～10mg test specimen is keeping at the temperature of 180～220℃ during this test. Oxidation reaction started when the antioxidant is exhausted. The time between starting point of oxygen injection and decreasing point of heat flow is OIT. Because the thermal flow curve decrease at the shape of exponent curve, it is not easy to select the exact point of OIT. We generally decide OIT at the cross point of baseline extension and exothermal slope line as shown on fig. 1.

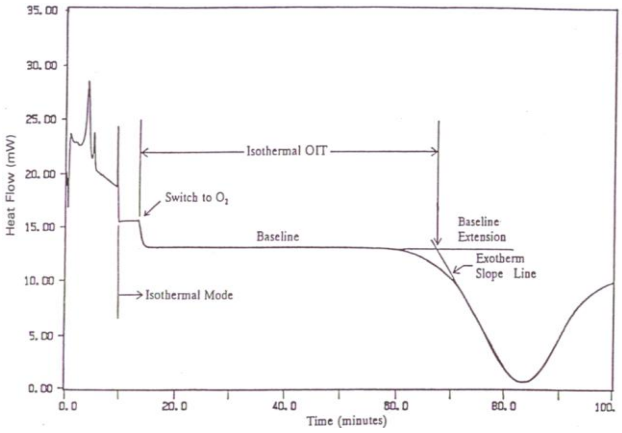

Fig. 1 General OIT curve

2.2 Problem of current OIT method

2.2.2 Low reproducibility

Because of scattered and non-linear curve as shown on fig. 2, precise reading of OIT was difficult. In case of double decreasing point as shown on point A and B on the fig. 2, it was not easy to select correct OIT point

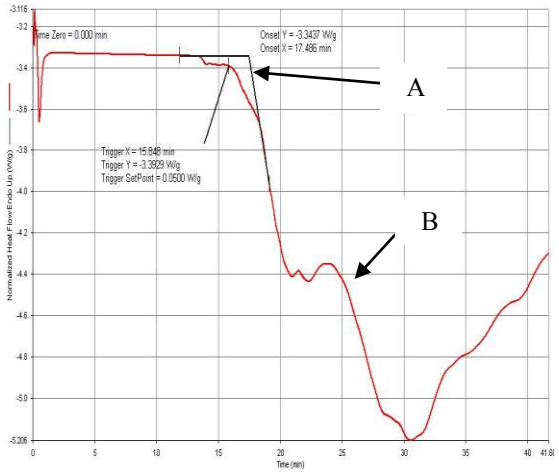

Fig. 2 Sample of bad OIT curve

2.2.3 No standard for OIT specimen

Because of no special requirement for specimen type, several types of specimens were used as shown on fig. 3. To evaluate the OIT accuracies of each specimen, statistics method was used. We performed 5 times OIT test for 3 kinds of specimens obtained from one material. It was found that standard deviation of OIT was 10.02 minutes. Reminding that OIT on fig. 3 was 17 minutes, standard deviation of 10.02 minutes was not negligible.

Fig. 3 picture of OIT test specimens

2.3 Improvement of OIT measurement

2.3.1 Improvement of OIT specimen

As result of brainstorming with several specialists, powder specimen was recommended to improve the accuracy of OIT measurement. Method of freezing at the liquid nitrogen and grinding was selected to make powder specimen of organic material. 0.1 mm powder specimen was prepared as shown on fig. 4.

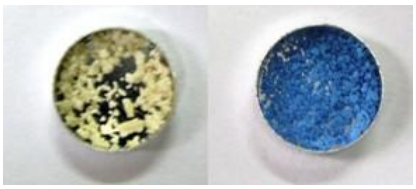

Fig. 4 Powder specimen for OIT test

2.3.2 Improvement of OIT curve reading

After improve the OIT test specimen, shape of OIT curve was significantly improved as shown on fig. 5. Since the OIT point had small radius, it was very easy to make cross line of baseline extension line and exothermal slope line. Standard deviation of OIT was improved to 1.38 minutes

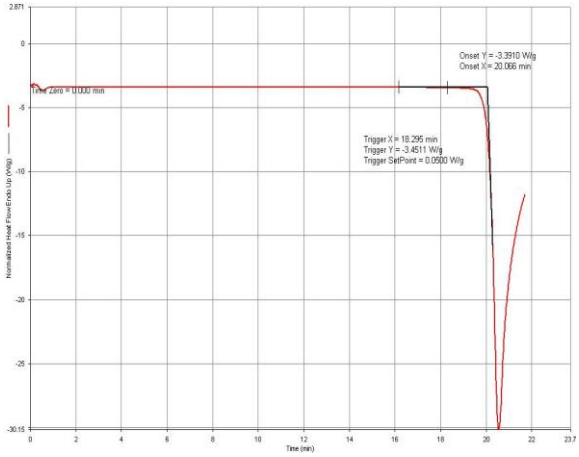

Fig. 5 OIT curve by improved test specimen

3. Conclusions

To improve the accuracy of OIT measurement, test specimen was changed from solid fragment to powder. Standard deviation of OIT was improved from 19.71 minutes to 1.38 minutes. Standard deviation was decreased 93%. This improvement of OIT accuracy is correspond to improvement of life evaluation accuracy from 19~54years to 2.5 years. It is recognized that powder specimen has to be used for best accuracy of OIT measurement.

REFERENCES

[1] EPRI, "Reduction of Oxidation Induction Time Testing to Practice as a Life Assessment Technique for Cable Insulation", EPRI-TR-106370-3427, 1996.
[2] Kyung-Heum Park, 'Application of OIT method for aging evaluation of NPP cable', Korea nuclear society spring meeting, 2006