ICEA Medium-High Voltage - Hot Creep / Solvent Extraction / Wafer Boil Tests

Why are the tests done?

In the 1970's the predominant insulation was thermoplastic polyethylene. At this time, thermoset (XLPE/EPR) compounds were becoming commercially available. To differentiate between the two, tests were needed to ensure that these compounds weren't being misrepresented in the industry. The tests were also applicable to semiconducting shields that were introduced later.

What do the tests tell us?

The Hot Creep / Solvent Extraction / Wafer Boil tests were all developed to determine if there was sufficient cross-linking in thermoset compounds such as XLPE, EPR, and TR-XLPE. Hot Creep and Solvent Extraction provide a quantitative assessment of cross-linking in two different ways, while Wafer Boil provides a qualitative measure of cross-linking. The current requirements in ICEA standards are : Wafer Boil for conductor and insulation shields, Hot Creep for Insulations, and Solvent extraction as a referee test if the insulation test fails.

Solvent extraction provides the most precise determination of cross-linking by yielding a value for % gel content. The test however, takes the longest to perform and isn't ideally suited as a routine production test. On the other hand, Hot Creep and Wafer Boil have evolved to become routine production tests that take much less time and provide more immediate feedback for process control on a CV line.

How is the test done?

<u>Wafer Boil</u>

A representative cross section containing the extruded conductor shield and insulation shield shall be cut from the cable. They may, or may not, be separated from the insulation. The wafers, or rings, shall be immersed in boiling decahydronaphthalene with 1% by weight antioxidant 2246 for 5 hours. The wafers shall then be removed from the solution and examined under a microscope for shield/insulation continuity. There shall be no breaks in the semiconducting shields, however, total or partial separation of the semiconducting shields from the insulation is permissible. Partial loss of the shields is also permissible provided each shield is a continuous ring.

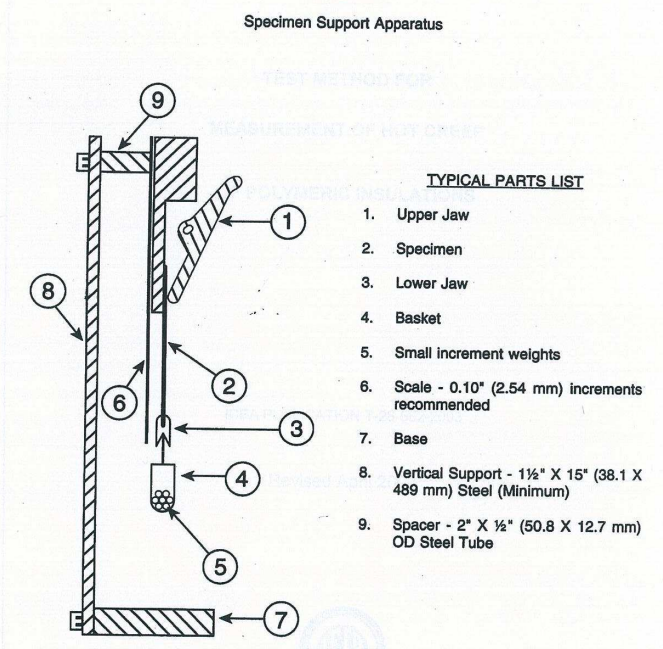
Hot Creep

The test comprises two parts – Elongation Test and Set Test.

Elongation Test : A specimen is subjected to a constant load stress while suspended in an air oven at a specified temperature for a specified time period. At the end of the time period, the increase in elongation of the specimen is determined.

Set Test : Immediately after the elongation test has been completed on a specimen, the same specimen with the load stress removed, will be subjected to an additional time period in the oven at the same elevated temperature. The specimen is then removed and allowed to cool. The set of the specimen, based on original length, is then determined.

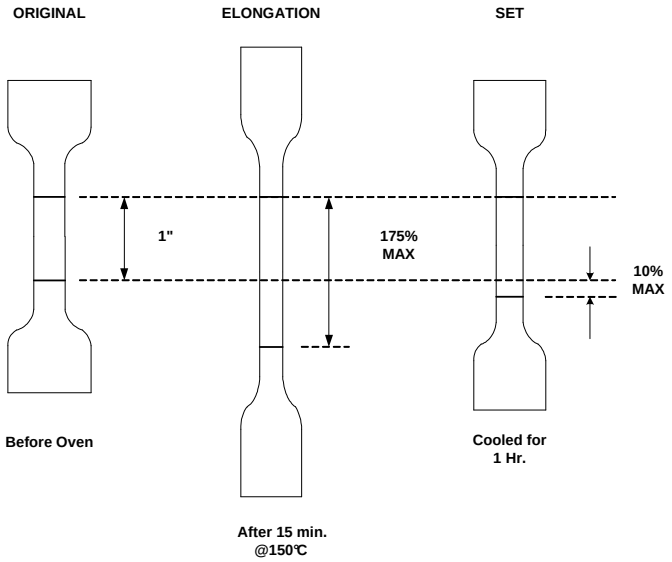
(Fig. 1)



The test consists of preparing a tubular specimen for insulations of 60 mils, or less, or a die-cut specimen for larger thickness insulations. For larger thicknesses the specimen shall be taken from the inner 25% of the insulation. The cross-sectional area shall be calculated to determine the necessary weight to be suspended from the specimen to achieve a 29 psi total load. The specimen shall be placed in an oven at 150 C . After 15 minutes exposure, without removing the specimen from the oven, the elongation is measured and a % change is recorded from the original 1” gauge marks. The Set Test is performed on the same specimen while in the oven, the load is removed and left for 5 minutes. It is then removed and allowed to cool to room temperature. The distance between the original 1” gauge marks is again measured now that the specimen is cool and % change to the original is calculated as the “set” value.

(Fig. 2)

Die Sample Results



For more detailed information on the test refer to ICEA T-28-562.

The criteria for passing the Hot Creep test is shown below in Table 4-2 from ICEA S-94-649 (2004).

ICEA S-94-649-2004 DATE: 10/14/04

Table 4-2
Insulation Physical Requirements

Physical Requirements	Insulation Type					
	XLPE and TRXLPE	XLPE Class III and TRXLPE Class III	EPR Class I	EPR Class II	EPR Class III	EPR Class IV
Unaged Requirements						
Tensile Strength, Minimum psi (MPa)	1800 (12.5)		700 (4.8)	1200 (8.2)	700 (4.8)	550 (3.8)
Elongation at Rupture, Minimum Percent	250		250			
Aging Requirements After Air Oven Aging for 168 hours						
Aging Temperature, °C ± 1 °C	121	136	121		136	121
Tensile Strength, Minimum Percentage of Unaged Value	75		75	80	75	
Elongation, Minimum Percentage of Unaged Value	75		75	80	75	--
Minimum Percent at Rupture	--		--	--	--	175
Hot Creep Test at 150 °C ± 2 °C	**Unfilled**	**Filled**				
*Elongation, Maximum Percent	175	100	50			
*Set, Maximum Percent	10	5	5			

*For XLPE and TRXLPE insulations if this value is exceeded, the Solvent Extraction Test may be performed and will serve as a referee method to determine compliance (a maximum of 30 percent weight loss after 20 hour drying time).

Solvent Extraction

This test determines the precise amount of cross-linking that has taken place in a polymer by measuring the gel content (insoluble fraction). The procedure involves grinding a very fine sample from the insulation and boiling it in a solvent that dissolves the un-cross-linked portion. The sample is then placed in a vacuum chamber and heated to drive off any residue of the solvent. The sample is then weighed. From knowing the weight before and after this process, the percentage of extractables can be determined (soluble portion). This test method serves as a referee test to the Hot Creep test with a maximum of 30 percent extractables (weight loss), or minimum 70% gel content, after a 20 hour drying time in the vacuum chamber.

Test Evolution

It was most likely the Solvent Extraction test that was used by the early developers of thermoset compounds. It first appeared in a power cable standard in AEIC No.5 in 1969 for insulation. With the introduction of cross-linked extruded semiconducting shields, the specification was then expanded to include these materials in 1974. It wasn't until the 1987 edition of AEIC CS5 where the Solvent Extraction test for extruded semi-conducting shields was renamed the Wafer Boil test with the same requirements as in previous editions. Also, the Hot Creep test replaced the solvent extraction test. Solvent Extraction testing took many hours to perform which could result in possibly thousands of feet of cable being produced which would have to be scrapped if the test failed. It was determined in the early 1980's that a faster test method was needed which could determine if the cable was sufficiently cured. Thus, the Hot Creep test was developed within ICEA by performing round robin testing of cable insulations with various states of cure. With good correlation between the two, Hot Creep replaced Solvent Extraction as the main production test.

The Hot Creep test was used for a short time in the 1994 AEIC CS5 standard to determine the cure state for semiconducting insulation shields. However, it was discovered that insulation shields could be formulated to meet hot creep requirements but have not been sufficiently cured when tested by the solvent extraction test. The use of the hot creep test was discontinued for insulation shields by ICEA standards and AEIC CS8 in 2000. This left the Wafer Boil test as the main test for this material.