論 文
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폴리에틸렌 내에서의 워터 트리의 구조와 전기적 특성

Internal Structure and Electrical Behaviour of Water Trees in Polyethylene

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요 약

워터니들둘을 사용하여 폴리에틸렌내에 워터트리를 형성시켰다. 이 트리의 전기적 특성은 도전체이기보다는 유전체의 특성을 지니고 있다는 것을 실험적으로 보여주었다.

한편, 이 트리의 구조는 서로 연결되지 않은 캐비티로 형성된 것으로 생각되며 이러한 결과는 워터트리의 단면을 잘라서 헬륨가스를 통과시킨 결과에 의해 검증되었다.

Abstract

It is shown that water tree grown using the water needle method in polyethylen do not behave as conductor but as dielectrics.

It is decuded that water trees are not non-interconnected cavities. This result is confirmed by gas flow measurements through freeze-fractured water trees.

1. Introduction

Despite numerous studies made on water trees in the last ten years the detailed mechanisms of tree formation are not yet understood. One of the reasons for this situation is the fact that the very nature of the matrial degradation is not well known. In particular, the question of the internal structure of the water trees, the knowledge of which is essential for testing several proposed theories has been much debated for a long time. It is known, of course, that water trees contain a large amount of water, that order of magnitude of the volumic water content being 2 to 10% of the water tree volume.1) This value is considerably higher than the usual solubility of water in polyethylene. We conclude that water molecules cannot be uniformly distributed but must be clustered in pockets. There may be spherical cavities or elongated one like channels. Opinions on the shape of these pockets are divided. Some authors consider that a water tree is made up of water-filled channels as suggested by observtion with a microscope. From scanning electron microscope photographs, it has recently been asserted that continuous channels do exist, with diameters between 0.1 and 3.5 μm.2)

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Other authors have observed serise of spherical cavities which do not seem to be connected. Again using a scanning electron microscope, a large number of cavities ranging from 0.8 μm to 9.8 μm with no evidence of channels interconnecting the cavities has been observed in treed regions on cross-linked polyethylene.[3] These differences in opinion reveal the difficulty of direct observation of the internal structure of water trees. These are due in particuler to the cross section and to the fact that the observed situation in the corss section may be different from the actual one present during the water tree growth.

We present here two indirect methods for studying the internal structure of water trees. The first is based on the relationship existing between internal structure and electrical behaviour : a water tree made of adjoining conducting channels should behave as a conductor whereas if it is made of non-interconnected conducting cavities it should behave as a dielectric. The second is based on measurements of gas flow through freeze-fractured water trees, from which continuous channels may be detected.

2. Experimental

A. Electrical Measurements

Water trees were grown via the water needle method using polyethylene specimens and a visualization system all of which have been previously descrided.[4] The system, using microscope, video camera and television enables us to observe continuously the same tree and to measure its length throughout its propagation even under electrical stress. Some modification of our usual specimens were needed, as shown in Fig. 1 to detect indirectly the variations of the electric field in polyethylene near the electrode facing the water tree during its growth. The voltage V between the water needle and the grounded electrode was divided into two parts which depend on resistance R and capacitance C between these two electrodes.

With the resistance R and the voltage V being constant, the possible variations of capacitance C due to the growth of the tree may be detected by the variations of voltage v across R.

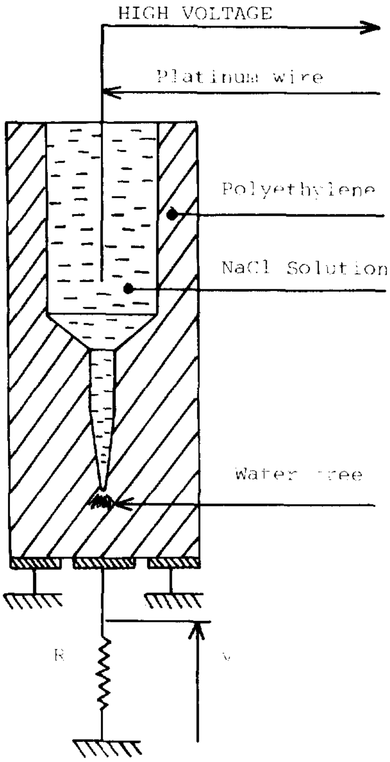

Fig. 1. Schematic drawing of the specimen

Experimental conditions were the following : radius of curvature of the water needle r=10 μm; distance between needle and plane electrode d = 1 mm: area of plane electrode A=5 mm²; resistance R=1 kΩ; voltage V=12kVpp; frequency f=1.5kHz. The material was LDPE free from additives and the liquid was an aqueous solution of NaCl with concentration 0.5M/ℓ Voltage was applied across a platinum wire in the liquid and silver paint on the opposite electrode.

B. Gas Flow Measurements

Large water trees, from 500 to 600 μm long, were grown in the experimental conditions described in section A. Then, water was removed, in vacuum at room temperature, and specimens were immersed in liquid nitrogen for several minutes before being freeze-fractured in order to get cross sections of the trees. Five sections were obtained at distances from 200 to 400 μm from the needle tip. A photograph of such a section is given in fig. 2. The sectionned end of each specimen was connected to a leak detector (LEYOLD-HERAEUS, model ULTRATEST M2 BY) and

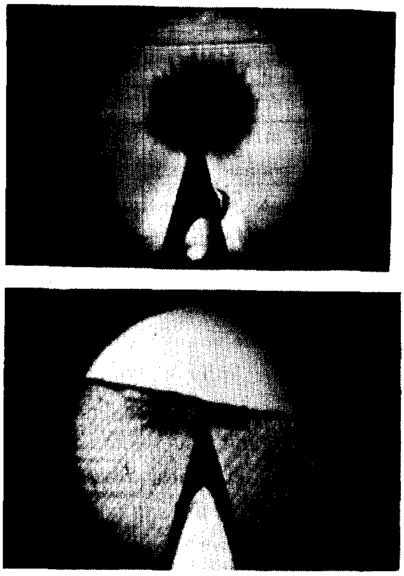

Fig. 2. Photographs of water trees
a-typical aspect of a water tree grown in our test conditions. Length of the water tree : 400 μm.
b-freeze-fractured water tree. The distance between water needle tip and the cross section is 200 μm. The length of the water tree was 500 μm.

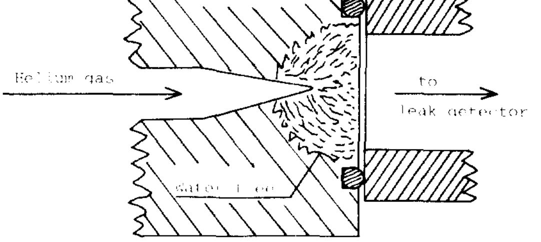

Fig. 3. Schematic drawing of the arrangement for gas flow measurement

helium gas was admitted at the other one (fig. 3).

3. Results and Discussion

The ratio of voltage v across resistance R with and without water tree v(L)/v(o) is given in fig. 4 (continuous line curve) as a function of the ratio L/Lo, L being the water tree length and Lo the distance between the tip of the needle and the opposite electrode. One notices an increase in v(L)/v(o) which reveals an alteration of the electrical behaviour of the material. In order to compare the electrical behaviour of

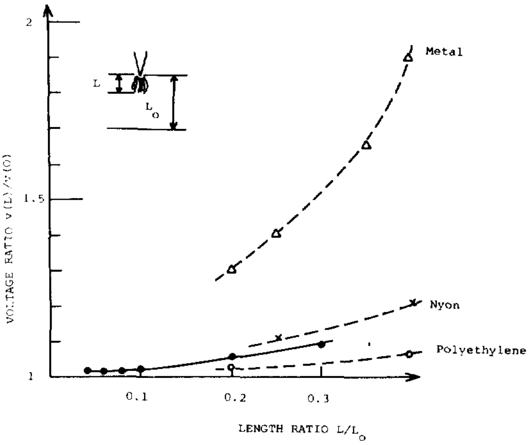

Fig. 4. Voltage ratio v(L)/v(O) with and without water tree(or model) versus water tree (or model) length L/L_0

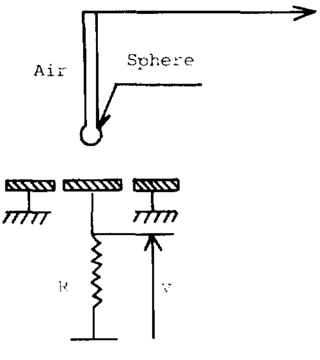

Fig. 5. Schematic drawing of the model

a water tree with those of a metal or of a dielectric we carried out additional experiments on models. Models of the actual specimen were made on a scale 10 times larger, with polyethylens beeing replaced by air and water trees by metallic or dielectric spheres of different radii L (fig. 5). The voltage ratio v(L)/v(o) was measured as a function of the radius L for metal, nylon (permittivity: $\varepsilon_r=3.3$) and polyethylene ($\varepsilon_r=2.3$). Results are presented in fig. 4 using dotted lines. One can see that the curve for the water tree is located between those for nylon and polyethylene, which shows that a water tree has the behaviour of a dielectric and not that of a conductor. The permittivity of the water tree can be estimated to be about the average value between 3.3 and 2.3 with respect to polyethylene, that is about 6 with respect to air.

Two important results can be inferred from this experiment. First, it has been proved in a direct manner that a water tree cannot be considered as a conductor. The knowledge of the electrical behaviour of water trees and of the local electric field which can be deduced is essential for the interpretation of the propagation curves. Until now, water trees had been considered as conductors in most major works, c.f. ref[5]. Filippini et al[4] departed from this theory. Secondly, it is shown that a water tree is not made of conducting adjoining channels. Indeed, if it were so. the capacitance of the tree would be that of a conductor having the same external appearance as the tree, but the experiment shows that it is not the case. Continuous insulating channels could exist, but, in the range of voltage, frequencies and conductivity of our experiments, a channel of 1μm diameter, considered as an elongated ellipsoid, behaves as a conductor up to lengths of a few hundred microns[6, 7] and so the assumption of insulating channels must be ruled out. Thus, it can be concluded that continuous channels do not exist and that the ionic solution is located in non–interconnected cavities. As our experiments are carried out under voltage, this result corresponds to the actual situation during the growth of water trees.

It would be interesting to examine correlations between the permittivity of the water trees and the size, shape, distribution and concentration of water inclusions in polyethylene. The results of this work do not allow an elaborate study on this subject. It can only be said that, using usual formulae for dielectric mixtures, a volumic concentration of 10% water in polyethylene, as observed in water trees, gives the right order of magnitude for the permittivity.

Gas flow measurements corroborate the results on the internal structure of water trees deduced from electrical measurements. Experiments were performed on 5 water trees cut at distances between 200 and 400 μm long we have calculated the possible helium flow in the conditions of our experiments and we obtained about 5×10^{-7} mbar. liter/sec. This value is more than 1000 times the measured one the order of magnitude of which rather corresponds to the diffusion of helium gas through polyethylene. The result shows that there are no continuous channels from one end of the sample to the other, excluding regions of the water tree located at distances lower than 200 μm from the needle tip in which interconnected channels might exist.

We believe that the situation observed in the samples is the same as the actual one during the growth of the trees for the following reasons : (1) the time interval between the switching off of the voltage and the gas flow measurement is about two hours. This time is too short to allow any molecular reorganization which might block possible channels. Strain relaxations would be possible but it seems unlikely that they could block all channels (if there were any) : (2) freeze fracturing contrary to other methods do not alter the material near the cross section.

Unlike our first experiment on the electrical behaviour of water trees, this latter experiment cannot prove that continuous channels do not exist but it gives one more argument in favour of this result. Its value is comparable with that given in[3] from scanning electron microscope photographs. Our results are in agreement with this last study, the interest of which, with respect to others using the same technique, comes from the fact that observations are taken on freeze fractured surfaces and on sections in two directions, perpendicular and normal to the tree axis.

4. Conclusion

Water trees grown in our test conditions do not behave as conductors but as mixed dielectrics made of polyethylene with water inclusions. They should not be considered as formed of adjoining conducting channels but of non-interconnected cavities.

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