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THE ELECTRONIC TEST FOR A DIAGNOSTIC OF CARBON BLACK DISPERSION IN THE RUBBER

Robert Jurik
Faculty of industrial technologies, Department of physical engineering of materials
Ivana Krasku 491/30, 02001 Puchov
Slovak Republic

Ivan Ružiak, Ivan Kopal
Institute of Materials and technological research, Faculty of industrial technologies
Ivana Krasku 491/30, 02001 Puchov
Slovak Republic

ABSTRACT
The paper deals with the ”in situ” in a rubber mixer measurements of alternating current conductivity (AC). The presence of carbon black (CB) in the mixer sufficiently changes the electrical properties of the blend. The mixing process was continuously monitored through changes of such physical quantities as resistance R, impedance Z and phase angle φ. The transient characteristics of these parameters reflect the CB dispersion in the rubber and they were continuously monitored. The very good repetition ability of mixing process was observed.
Keywords: Ac conductivity, carbon black, mixing process

1. INTRODUCTION
Process of vulcanization starts after the mixing of chemical components with different chemical activity but it is strongly influenced in all cases by very high viscosity of natural or synthetic rubber [1]. Specific conditions of mixings process are also followed from the presence of fillers of different chemical quality which is in details described in the work [2].
The process of carbon black mixing is assumed in following steps. After the addition of the filler the agglomerates of the size up to 10-100 micrometer are created. In the blend the rubber shells are created around the filler particles followed by filling up the voids within the filler agglomerates. Under stretching deformation caused by shearing forces aggregates of 100 nanometers till 0,5 micrometer appears after breaking of agglomerates. Primary particles as a final product of mixing have the dimension of approximately 20 nm. Smaller aggregates and primary particles appear on the expense of larger aggregates and agglomerates. It is necessary to underline that the whole process of mixing in the period of a mastication and plasticization is accompanied by changes of rheological properties, where the viscosity decrease is caused by both polymer breakdown and temperature rise respectively. The viscosity fall reach the plateau with the smallest particle size (aggregates) in the structural point of view. Further mixing homogeneously spreads the particles in the blend volume only.
The quality of mixing process depends on the ability of mixer and physical and chemical condition of mixing process to create the best dispersion as it is possible.
A quality of the mixing process is in some cases judged by measurement of Payn’s effect (characterized by decreasing of Young’s modulus with increasing of sample loading) after the sample preparation [2].

Other way how to identify the processes running over in the mixture is to measure the changes of electrical conductivity. The influence of molecular structure of the rubber matrix on the carbon black dispersion in rubber compounds has been characterized using the method of the online measured direct current electrical conductivity during the mixing [3].
The change of the conductivity and carbon black dispersion are determined by a very complex behaviour of the mass in the mixing chamber. A corresponding change of the online conductivity, the rubber infiltration into the carbon black agglomerates, and the carbon black dispersion value in all the systems investigated have been observed on large scale of rubber mixtures. A linear chain structure as well as the low molecular weight of the matrix rubber accelerates the infiltration process and hence the carbon black dispersion process takes place faster. A lower chain mobility caused by a modification of the rubber with functional groups leads to a deceleration of the carbon black infiltration.
In this paper we are presenting results of the electronic „in situ“ test of rubber blend mixing. It has been tested changes AC electric parameters such as impedance, capacity, phase angle caused by addition of chemical components to the natural rubber.

2. EXPERIMENTAL PROCEDURE

The rubber blends were mixed in two steps. In the first step we started with the mixing of natural rubber, then we added ZnO, then carbon black and finally oil. As a filler we used used carbon black N660 Cabot. The temperature of the Brabender plasti-corder PLV 151 chamber was maintained at 80 degree of Celsius. In the second step, sulphur as a vulcanization agent was added to the mixture. The vulcanization temperature was 150 degrees of Celsius.
The mixing process has been continuously monitored (Figure 3.) by the capacity probe located in the mixing chamber of the mixer. Sensor was connected with RLC bridge Fluke Pm 6306controlled by computer. Values of R, Z*, and phase angle were continuously monitored. Z*,R and changes were continuously displayed on the computer screen as a function of mixing time. The cylindrical capacitor as a sensor was located in the middle of the chamber, near the mixing blade to obtain the continuous contact of the blend with the sensor.

3. RESULTS AND DISCUSSION

In the first step we have tested the sensitivity of chosen electrical AC circuit parameters caused by chemical agents added to a natural rubber. In the Figure 1. the dependence of the impedance and capacity versus time for the sample 1 is plotter. The same parameters are plotter in the Figure 2 for other sample of the same chemical composition. Both pictures show a characteristic impedance fall after the addition of the carbon black to the natural rubber. On the other hand in the same time it is observed the capacity peak probably caused by immense increase of the relative permittivity and corresponding sensor capacity. In both pictures there is visible also the peak corresponding to the oil addition (see incorporated frame with chemical processing inside the picture). Comparison of both plots shows a very good repetition ability of the used testing method.

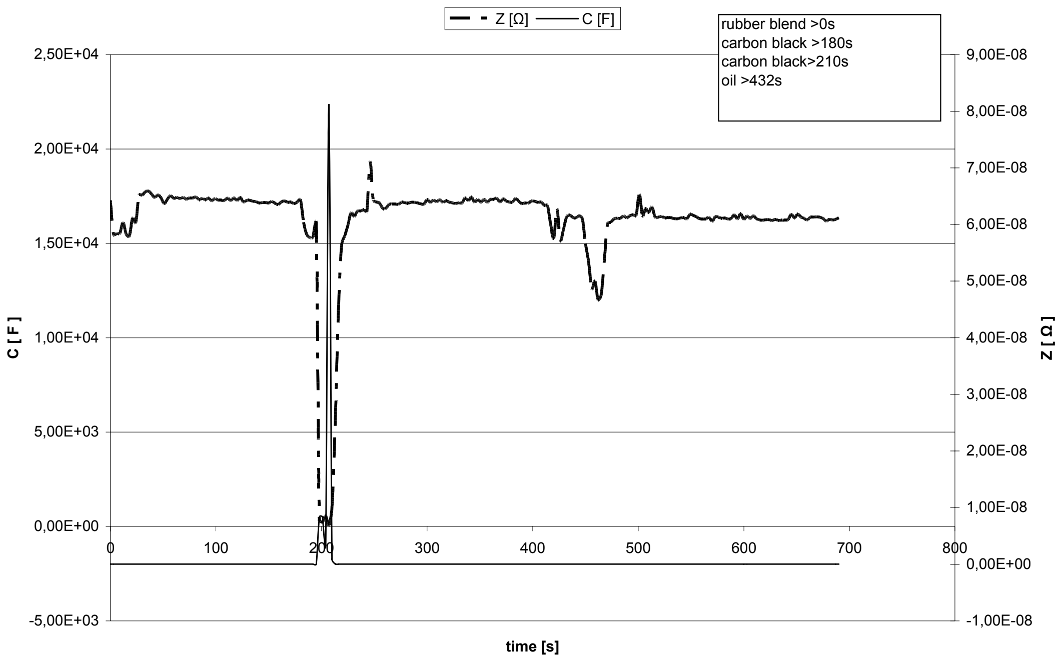

Figure 1. The dependence of the impedance and capacity versus time for the sample 1

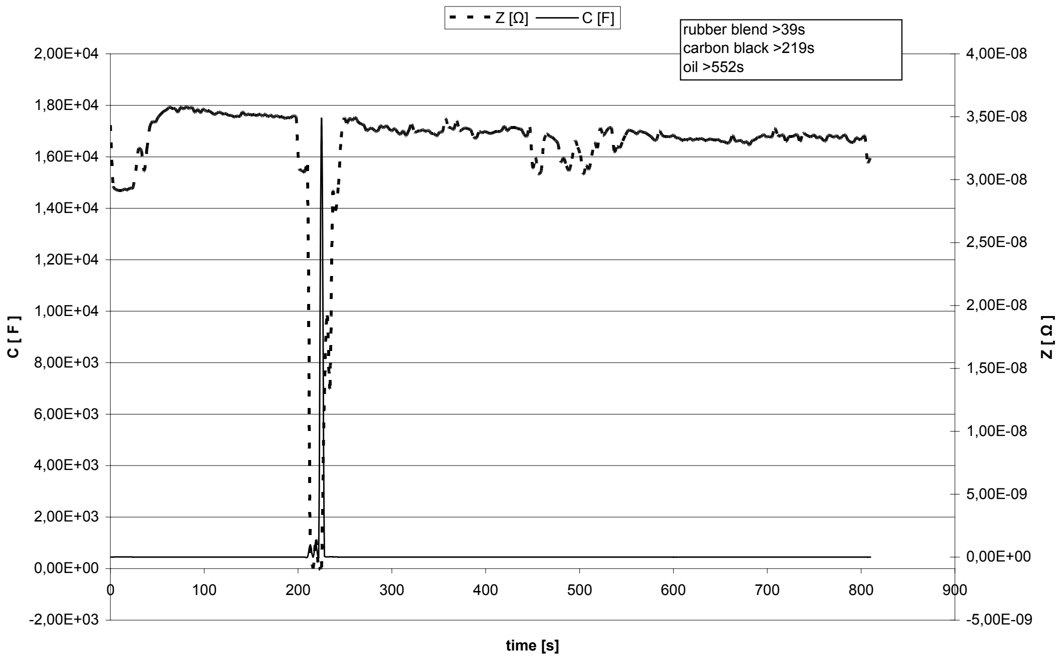

Figure2. The dependence of the impedance and capacity versus time for the sample 2

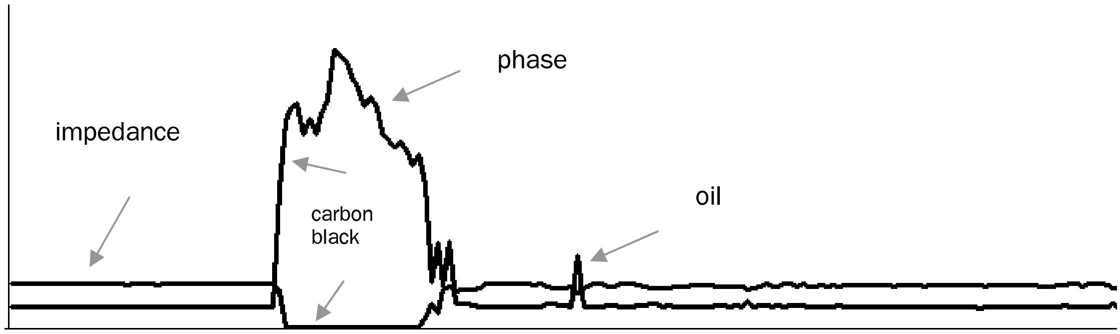

Figure3. Online monitoring on the computer screen for the sample 2

4. CONCLUSION

In the contribution we presented the results of the apparatus with capacity sensor for the detection of dispersion of carbon black in natural rubber. We obtain very good repetition ability and sensitivity to the presence of chemical a agent reflected in changes of AC circuit parameters such as impedance and capacity.

5. REFERENCES

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