SAFETY AND TRANSPORT
ELECTRONICS

WP2 SAMPO
– Online condition monitoring techniques - Dielectric Properties

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RISE Report :

Content

Background

Changes in the chemical structure and overall composition of the materials are likely to affect the dielectric properties and there are previous examples where changes in dielectric properties have been linked to aging of polymeric materials (Daily, 2015; Li, 2011). The effects of ageing on the dielectric behaviour of the materials in question could be measured using e.g. impedance/dielectric spectroscopy. A very convenient method to monitor the status of rubber or polymer materials would be to measure material changes online. If there are large enough changes in dielectric behavior of the materials that can be directly related to the ageing process, it should be possible to follow these changes using antenna dielectric sensors (Huang, 2015). By broad band frequency mapping of the dielectric behavior of the materials under test, more narrowband antenna like structures could hopefully be designed to fit the frequency providing the highest sensitivity and ease of use at lower cost. Placement, environmental factors and calibration of sensors would likely also be issues necessary to address as well as monitoring moisture and temperature to avoid overlapping effects of moist content and degradation of the monitored polymers. This strategy towards assessment of polymer ageing has been part of Task T2.1 - Online condition monitoring techniques in the SAMPO project.

Goal of the study

The goal of this study was to assess if the accelerated ageing performed within the project induced measurable dielectric changes in some of the rubber materials and, if possible, identify specific frequency regions with more pronounced as well as systematic changes. We also wanted to investigate the feasibility of the concept of online monitoring of dielectric changes in similar rubber materials over extended periods of time.

Methods

As the frequency region of interest is unknown for any new material under investigation a broad band measurement method is needed. For this reason, a coaxial probe was chosen for the dielectric characterization of the materials. The coaxial probe is in principle a monopole antenna sensor, albeit not the type of antenna sensor we hope to see implemented as a result of this work. Instead of incorporating the antenna in or on the material the dielectric probe is pressed against the material. This approach facilitates measurements on several samples without extra fabrication steps, but also introduces some uncertainty in the quality and consistency of the contact between the probe and the material under test.

Measurements on aged samples

Measurements have been performed on provided EPDM samples that were cut out from the same piece of test material after which they were put through accelerated ageing to different degrees. The shape and thickness of the samples were not optimal in regard to compatibility with the dielectric probe method. They did e.g. not appear to quite fulfil the condition of being semi-infinite (thick enough to not measure out the other side of the material) and there were some difficulties in ensuring a uniform contact between sample and probe. For this reason, the absolute values of the measured data should not be taken as the true values for the permittivity of the materials. However, we believe that trends in changes can be taken as indicative if they are systematically detected over both several measurements at different sites on the same sample as well as between different samples of the same type.

Measurements were performed for three unaged samples and four samples aged at 140 °C for 6 months, of which two were also irradiated in cycles between ageing. Efforts were made to also include a sample that was aged in the microcalorimeter at 60 °C for 21 days, but the shape and size of the sample made it difficult to get consistent results, which is why we chose not to include it in this report. The probe was disconnected from the samples and then reconnected between measurements to ensure measurements were performed at more than one specific sample location. This also helps in ruling out that the variation in results between samples could simply be due to differences in the contact between probe and sample.

Before the first measurement the cable was placed in a fixed position to the extent possible. The measurement set-up was then calibrated for the entire measured frequency spectrum using air, milli-Q water and a short as reference points. The placement of the measurement equipment unfortunately turned out to be sub-optimal and it is difficult to rule out cable drift due to other activities in the vicinity of the measurement set-up. This may also be one reason why it was difficult to realize a good calibration for the lower frequencies.

Online measurements

Online measurements were performed on a sample placed in a climate chamber for accelerated ageing.

The measurement probe was inserted into a climate chamber set at 50 °C. This temperature was chosen as it was the highest temperature for which there appeared to be no risk of damaging the measurement probe available to us. The probe was calibrated in position, at the intended measurement temperature, with air, milli-Q water and an electrical short as references (references also heated to the measurement temperature). The probe and sample were pressed together and fixated, using a sample holder made of PTFE, to achieve good and consistent contact between probe and sample throughout the measurement cycle. The Vector Network Analyzer used to sweep and record the frequency response of the probe and sample was programmed to perform one measurement sweep approximately every hour for 1001 hours, resulting in a total measurement/ageing cycle of a little more than 44 days.

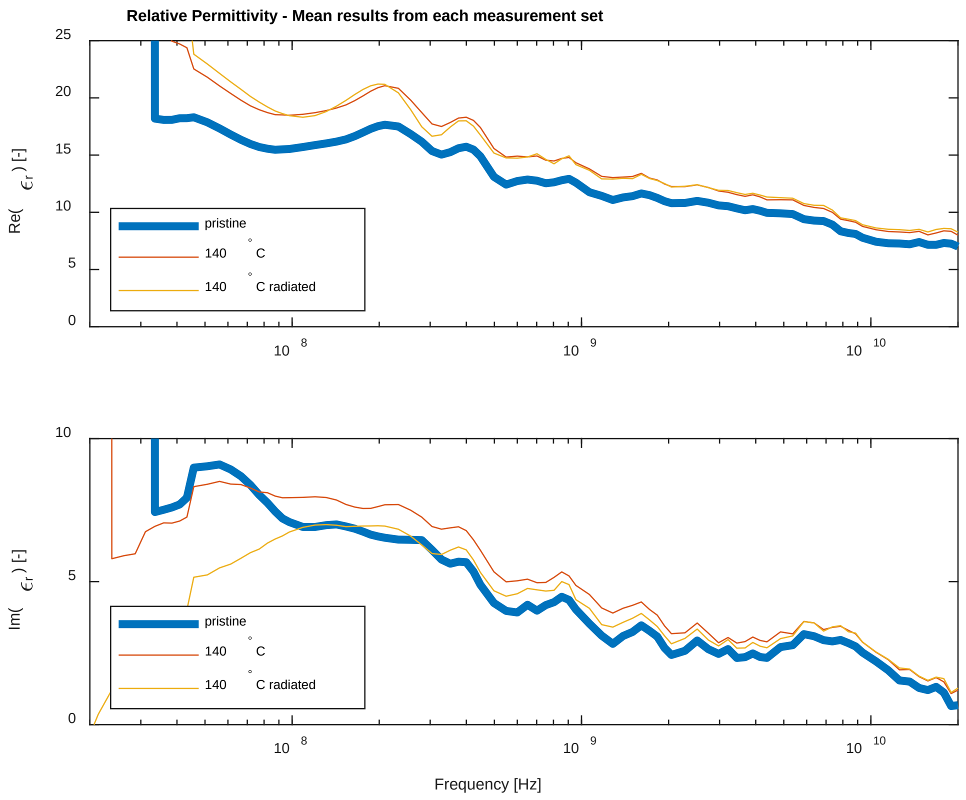
Results and discussion

As the wavelength becomes shorter with increased frequency it is more likely the samples could fulfil the semi-infinite thickness condition at higher frequencies. The results from these frequencies should thus be considered more reliable. We also had more difficulties achieving a stable calibration at lower frequencies and for these reasons we will focus more on the results at higher frequencies for our analysis. This does not necessarily mean that those frequencies would be more sensitive to material changes, and thus better suited for sensing the changes, but thicker samples and a better measurement set-up would be needed to draw further conclusions about the lower end of the spectrum.

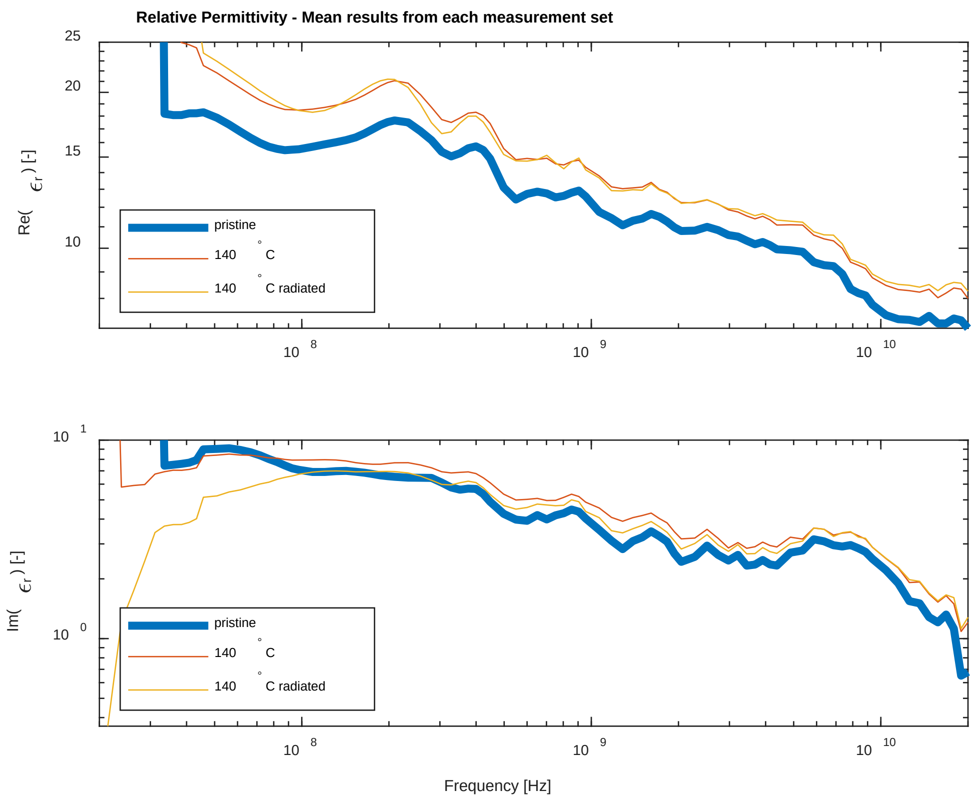
The overall quality of the measurement conditions and thus measurements was unfortunately unsatisfactory. As such the results should thus be seen as indicative more than scientifically sound.

Measurements on aged samples

A simple mean over all measurements made on each sample/sample group is presented below. Material pre-irradiation appears to have very little effect on the results for the samples aged at 140 °C. This is consistent with the results from the mechanical studies. A slight increase in the real part of the permittivity could indicate an increased density of dipoles. There appear to be no clear frequency shifts for the peaks of the imaginary part of the permittivity. Without knowing the exact blend of the rubber material under test it is difficult to make any statement on if the results are plausible or not.

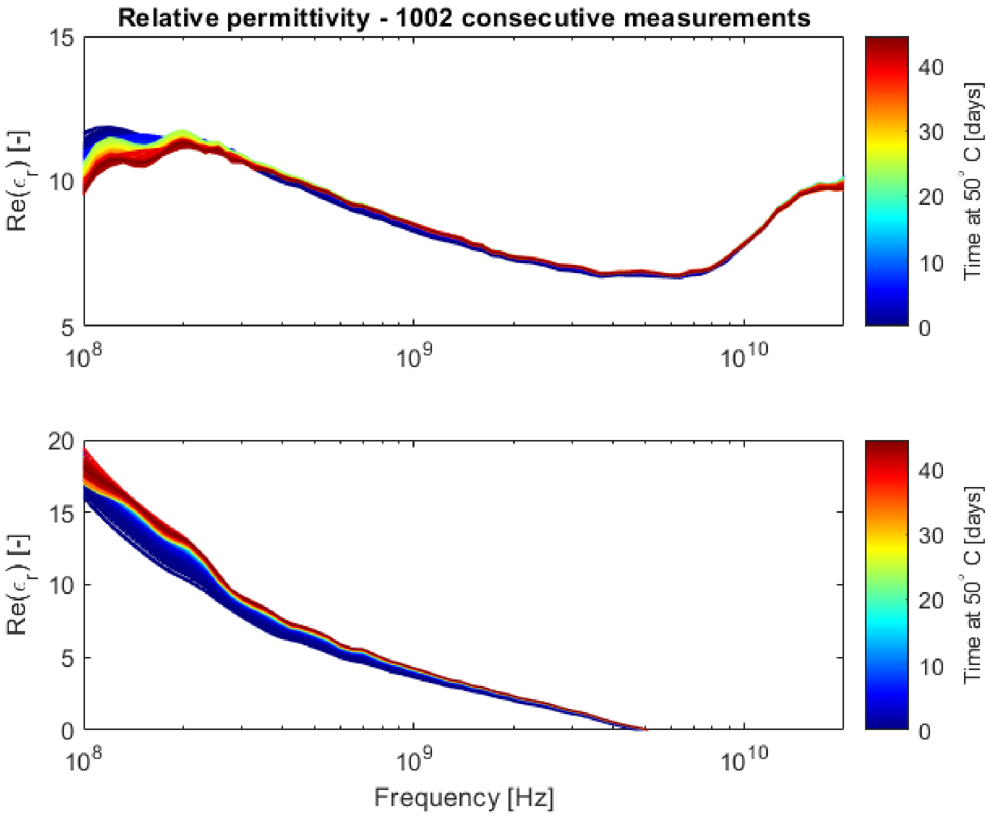


Looking at the data on a log-log scale there appears to be a shift in the dominant polarization mechanism at 6-10 GHz for this specific material, seen as a more pronounced reduction in the real part of the permittivity over those frequencies as well as what looks like a corresponding peak in the imaginary part of the permittivity in that same frequency interval. If the changes seen in the data is in fact due to the ageing mechanisms it appears that the ageing at higher temperatures affect the entire spectra, while the lower temperature aging only appears to affect the lower frequencies. This could indicate a possibility to use different frequencies for identification of different types of ageing.



Online measurements

The measured relative permittivity as function of frequency taken approximately every hour for a total of 44 days was plotted together with the colour axis indicating progression in time [days]. In most of the investigated frequency span it appears that the relative permittivity increases as time progresses. At this timescale the change is quite small, and the rate of change is highest at the start of the measurement series. The overall trend appears to be consistent with the results observed for the samples aged at 140 °C although the change seen at this timescale might be too small to draw any definite conclusion. At around 5 GHz the imaginary part of the permittivity appears to go below zero. This is likely not a true result, but rather an effect of reaching a Fabry-Perot resonance as the sample at this point is ¼ wavelength thick (~5-6 mm). The measurement set-up used here is thus not valid at these frequencies. Measurements on samples of a different thickness would be helpful to realize correct characterization over the entire spectrum.



Conclusions

Measurement results achieved can be seen as indications that the permittivity might prove a useful indicator for aging monitoring of some polymeric materials. To better determine the effect of ageing on the permittivity of the material measurements either need to be performed over very extended periods of time or at higher temperatures for a more accelerated ageing process. For this to be possible it would be beneficial to acquire a probe and/or probe system which is more robust and may function at higher temperature and have better handling of cable drift and sample contact variations.

As it has not been possible yet to identify any specific frequency region of interest, for the materials provided, frequency specific antenna sensor design and simulation has been postponed.

References

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