New Approach for Temperature Characterization of Low Loss Dielectric Materials

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Abstract. This article presents a new technique for the characterization of low loss dielectric materials at high temperatures: the split cylinder resonator cavity. This technique allows the accurate characterization of the dimensional and electrical characteristics of the cavity and the electromagnetic properties of a dielectric material, simultaneously in one temperature cycle. To do this, the couple $TE_{011}+TE_{013}$ is used to characterize the sample and the couple $TE_{012}+TE_{014}$ is used to characterize the cavity conductivity and dimensions. Measurements of conductivity and dimensions using the couple $TE_{012}+TE_{014}$ for a loaded cavity at ambient temperature and during a thermal cycle show an agreement with the values obtained using the couple $TE_{011}+TE_{013}$ for an unloaded cavity at ambient temperature and during a thermal cycle. The characterization of an Alumina sample during a thermal cycle performed using the new technique and the traditional one shows a good agreement for the permittivities and the loss tangents.

Keywords: Split cylinder resonator, characterization, substrates, dielectric, EM monitoring, temperature.

1. Introduction

The increase of frequency in microwave devices led material manufacturers to suggest a new range for low loss dielectrics. The study of the dielectric properties (ε' and tanδ) of these materials is a very active domain in material science, solid state physics, and electronics engineering. The accurate knowledge of these properties is extremely important, for two main reasons: first, these properties allow identifying the material's main physical properties, and second, because they have to be controlled for the applications that are very sensitive to their variations. The values of the dielectric characteristics of the material depend on the measurement frequency as well as the applied temperature [1].

Different microwave measurement techniques have been developed to characterize a dielectric sample at different temperatures: the resonant cavity technique, the microstrip line technique, the near field technique, the coaxial probe, the split cylinder cavity, etc. [1].

The method of the split cylindrical cavity is traditionally used to characterize accurately isotropic and homogeneous dielectric substrates having low and very low dielectric loss up to millimeter wavelengths. These cavities operate on resonance modes $TE_{01(2q+1)}$ which have a maximum electric field at the center of the cavities as shown in Figure 1(a) and 1.b. The electric field is slightly disturbed by the presence of the slot (under certain conditions of thickness and permittivity of material [2]) due to the single azimuthal component of the electric field of these modes. The temperature measurements are also possible as long as the conductivity and the dimensions of the cavity are known, depending on the temperature (typically from -20 to 80 ° C).

The characterization procedure consists of measuring the frequency of the TE_{011} and TE_{013} modes as well as the unloaded quality factor of one of these two modes. These initial steps are used to precisely determine the dimensions of the cavity as well as the equivalent electrical conductivity of the walls of the cavity. The loaded measurement, once the sample is introduced into the cavity (Figure 1(c) and 1(d), of one of the preceding two resonances which presents a frequency shift and a reduced quality factor, allows to characterize the complex permittivity of the sample at discrete frequency using a rigorous analytical model [3].

For temperature measurements, the same process is applied at each temperature point once the temperature equilibrium is attained. This involves two thermal cycles, the first is with an empty cavity, and the second is with a loaded cavity. This process is very long and requires a regular measurement of the resonance frequencies to observe its stabilization, synonymous to thermal equilibrium.

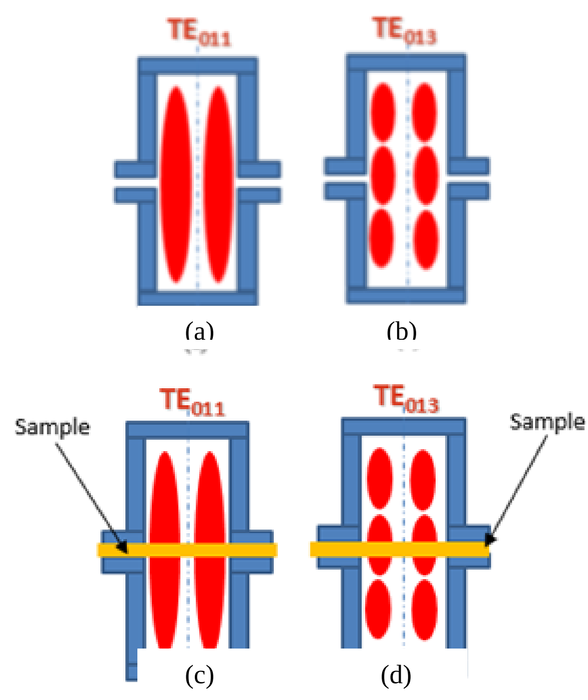

Fig.1. (a) TE_{011} mode unloaded cavity, (b) TE_{013} mode unloaded cavity, (c) TE_{011} mode loaded cavity, (d) $TE_{013,}$ mode loaded cavity.

In this paper, a new temperature measuring platform is presented. This platform allows reducing the number of measuring thermal cycles by carrying a simultaneous and real-time control of the dimensional and electrical characteristics of the split cylinder resonator (SCR) and those of the sample by avoiding the use of temperature models.

2. Principle of characterization

The solution presented in this paper is to be able to control, in the same thermal cycle, the characteristics of the cavity (actual dimensions and electrical conductivity of the walls) and those of the material. Using the even longitudinal modes $TE_{01(2q)}$ variations which are not sensitive to the presence of the dielectric sample (as shown in Figure 2(a) and 2(b), due to the null electric field at the center of the cavity where the sample is placed, it becomes possible to control the parameters of the cavity apart from those of the material.

Electromagnetic simulation based on 2D finite element method on free oscillations has been realized using EMXD Software. The free oscillation was used due to the low coupling that will be used next in the measurements; the EMXD software was selected due to its accurate results [4] compared to the available commercial software. The results have confirmed the low disturbance caused by a material for the TE_{012} mode (Figure 3) and TE_{014} mode (Figure 4), under certain conditions of sample thickness and permittivity. For the TE_{012} mode, the maximum frequency shift is 100 kHz for a sample thickness of 0.5mm, and the maximum quality factor degradation is 0.08%. For the TE_{014} mode, the maximum frequency shift is 600 KHz for a sample thickness of 0.5mm, and the maximum quality factor degradation is 0.5%.

The very small changes in resonance frequency and quality factor of the TE_{012} mode have an impact in the order of 0.1% on the permittivity and 0.16% on the loss tangent for an alumina sample of a thickness of 0.5mm for example. For the TE_{014} mode, the small changes in resonance frequency and quality factor have an impact in the order of 0.6% on the permittivity and 0.8% on the loss tangent of the same sample.

The electric field of the TE_{014} mode sees a little more the material because the variations observed for the quality

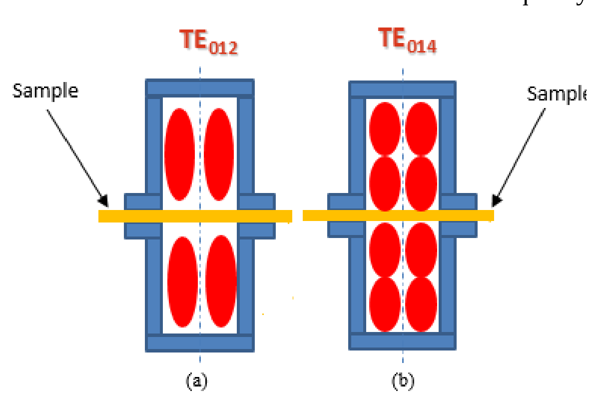

Fig.2 (a) TE_{012} mode loaded cavity, (b).TE_{014} mode loaded cavity.

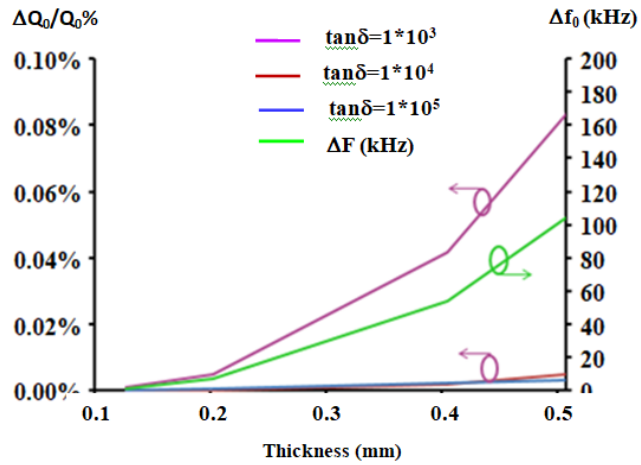

Fig.3. Variation of quality factor and the resonant frequency of the TE_{012} mode for an alumina sample (ε_r=9.8).

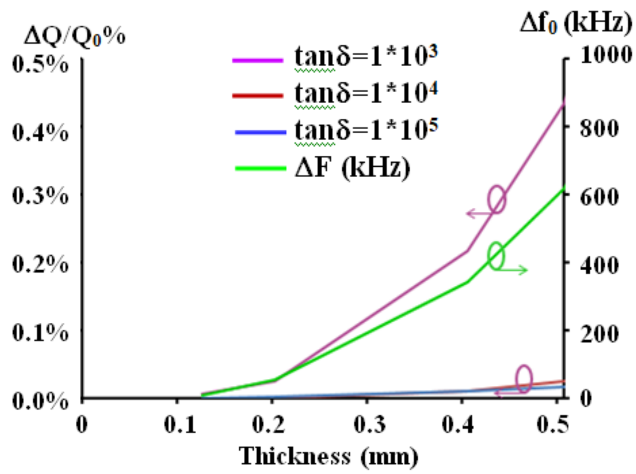

Fig.4. Variation of quality factor and the resonant frequency of the TE_{014} mode for an alumina sample (ε_r=9.8).

factor and frequency are multiplied by approximately 6 relative to those observed in the TE_{012} mode. These results were confirmed by measurements of some materials less than 0.5 mm thick.

3. Experimental dispositive

Recent vector network analyzers (VNA) with interpolated calibration allow, during a change of frequency band, to maintain the calibration procedure during the broadband characterization thanks to several TE01(2q+1) modes used [5, 6, 7, 8]. Indeed, the quality factor measurement, made in a narrow frequency band for accuracy considerations, no longer requires a specific calibration for each used band. Furthermore, the measurement accuracy of the quality factor can be improved by the use of a vector method [9]. Fig. 5 shows the experimental device. The SCR of diameter 45mm and height of 71.6mm is placed in an oven, the resonance frequency of the fundamental mode of this cavity TE_{011} (measurement frequency) is 10GHz. The sample that will be characterized is inserted into the slot at the middle of the cavity. In this case, the sample divides the cavity into two closed areas, avoids the gap between the sample and the cavity and at the same time cancels the effect of the sample expansion due to the temperature. The excitation of the TE_{01q} modes of the cylindrical cavity is achieved using two magnetic loops. These loops are arranged horizontally (parallel to the plane *r* and Θ of the cavity). A low coupling was used (-30dB) to obtain an unloaded quality factor equal to a loaded one. The waveguides outside the study can be cooled to ensure an ambient temperature at the vector network analyzer (VNA) accesses or the millimeter heads in order to increase the frequency of the characterization device. A thermocouple is connected to the cavity to measure its temperature. A special software was developed using Labview for the data acquisition and monitoring (the temperature of the cavity and the resonance frequency from the VNA).

The measurement of the S_{21} coefficient at the resonance frequency gives access to the coupling and thus to the unloaded quality factor considering an identical coupling at the two accesses. This value of S_{21} varies over 2dB during the thermal cycle. To control the origin of this variation, measures of cables and magnetic probes have been carried out. The used coaxial cables have good stability up to 200 ° C and induce an additional attenuation of about 0.05dB at 80 ° C on the modulus of S21. Magnetic probes made of semi-rigid cable introduce additional attenuation of 0.07dB.

4. Results

An acquisition and data processing software can take, at every selected Δt, the resonance parameters of each mode (TE_{01q}, q = 1 to 5) and characterize the conductivity of the OFHC copper cavity from the well-known analytical expressions of equations 1 and the actual dimensions of the latter (equations 2).

$$\sigma = \pi \mu_0 f_0 \left(\frac{Q_0}{K}\right) \quad (1)$$

$$K = \sqrt{\frac{\mu_0}{\varepsilon_0}} \frac{\left(x_{01}'^2 + \left(\frac{p\pi}{2}\right)^2 \left(\frac{2.Rc}{Hc}\right)^2\right)^{\frac{2}{3}}}{\left(x_{01}'^2 + \left(\frac{p\pi}{2}\right)^2 \left(\frac{2.Rc}{Hc}\right)^3\right)}$$

$$Hc = \frac{c}{2}\sqrt{\frac{p^2-q^2}{f_p^2-f_q^2}} \quad Rc = \frac{c.x_{01}'}{2\pi}\sqrt{\frac{q^2-p^2}{q^2 f_p^2 - p^2 f_q^2}} \quad (2)$$

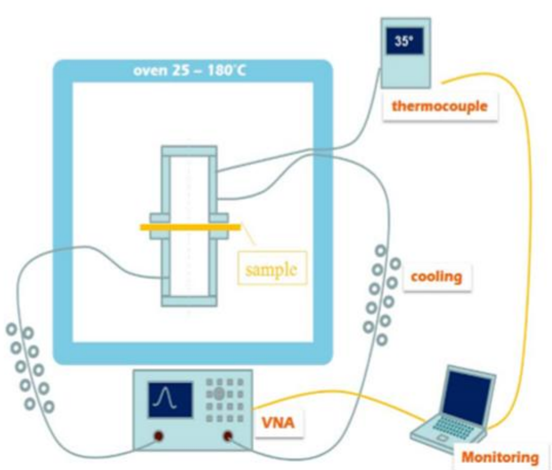

Fig.5. Temperature test device.

where x'_{01} is the first zero of the derivate of Bessel function $J_0(x)$, Q_0 is the unloaded quality factor of the considered resonance mode, f_0 its frequency, Rc and Hc are the radius and the height of the cavity and f_p and f_q the resonance frequencies of TE_{01p} and TE_{01q} modes, with p and q integers with p≠q. Fig. 6 shows the variations of the equivalent conductivity of the cavity at the ambient temperature obtained for five different modes (q = 1 to 5) and several hundreds of repeated measurements (> 360). The average of the conductivity is 50.4S/µm. An unloaded thermal cycle and a loaded one with a sample of alumina with a thickness e = 0.376 mm were performed to control the equivalent conductivity obtained by the TE_{012} mode during these cycles (Fig. 7).

The conductivity measured by the TE_{012} mode, with a loaded cavity, is identical to that measured when the cavity is unloaded. The evolution of metal losses can be controlled independently of the dielectric losses of the material during the thermal cycle. Repetitive measurements allowed determining a variation of ± 80kHz to ± 125kHz of the resonance frequency according to the measured mode.

These uncertainties, depending on the material used and the conditions of measurements, induce errors in determining the dimensions of the cavity. From the frequencies of the TE_{011} and TE_{013} modes, uncertainties of ± 0.23 µm on the radius and of ± 1.1 µm on the height of the cavity were observed.

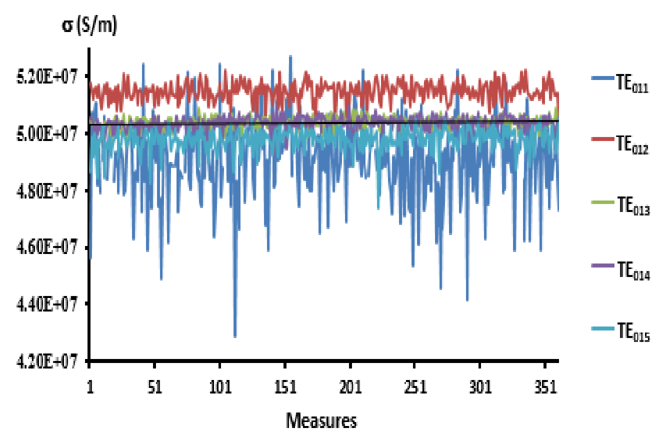

Fig. 6. Extraction of the equivalent conductivity of the walls of the unloaded cavity from TE_{01q} modes (q = 1 to 5)

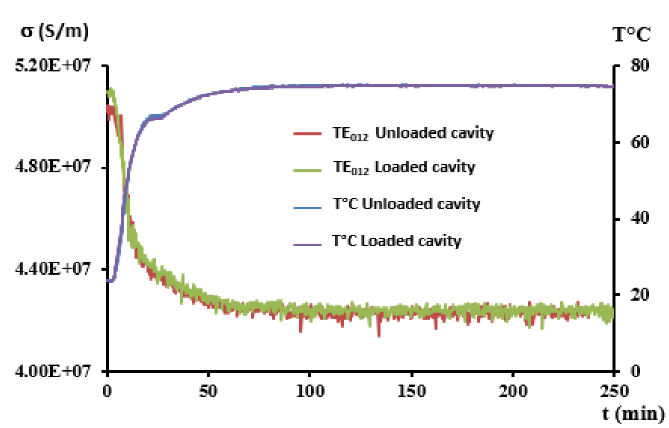

Fig. 7. Extraction of the conductivity of the unloaded and loaded cavity (Al_2O_3, e = 0.376 mm) in function of the thermal cycle.

These dimensional uncertainties are negligible for the determination of the complex permittivity of the considered samples (Fig. 8). Using the TE_{012} and TE_{014} modes, the same dimension uncertainties are obtained but the mean values are shifted by 3 µm for the radius with respect to previous calculations. These small offsets lead, for example for the considered alumina, to a shift of about $\Delta\varepsilon'/\varepsilon' \approx 0.2\%$ and $\Delta\tan\delta/\tan\delta \approx 0.1\%$.

During the heat cycle, the cavity expands and the new dimensions can affect the characterization of the material. A temperature measurement is performed for an unloaded cavity and a loaded one using two couples to experimentally estimate the expansion of the cavity.

Fig. 9 shows the variation of the radius and the height of the unloaded and loaded cavity during a thermal cycle. For an unloaded cavity and using the couple $TE011+TE_{013}$ (red circle in Fig. 9), the radius and the height of the cavity increase linearly. The observed expansion induces a 1.9% increase in permittivity and a 2% decrease in the loss tangent in the characterization of the reference alumina.

By determining the dimensions of the cavity during the loaded thermal cycle with the TE012 and TE014 modes (solid line in Fig. 9), we observe that the extracted dimensions using this couple are very close to the dimensions extracted from the TE011 and TE013 modes (difference between dimensions are less than 2µm).

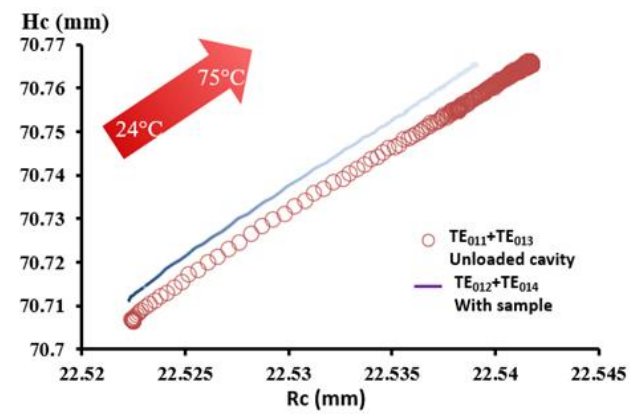

Fig. 8. Extraction of the dimensions of the cavity (unloaded) in function of two pairs of resonance modes.

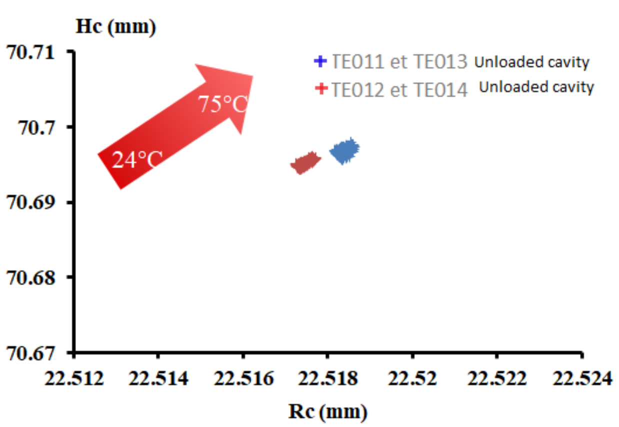

Fig. 9. Extraction of the dimension of the unloaded and loaded cavity in function of the temperature.

A microwave characterization of a sample using the new technique was proposed in this paper and a comparison with a traditional technique was realized. As stated before, and due to the variation of the complex permittivity (relative permittivity and loss tangent) in function of frequencies, one of the two resonances TE_{011} and TE_{013} is selected to realize the measurements.

Fig. 10 and Fig. 11 show a comparison between the complex permittivity obtained by the new technique and the traditional technique used for the temperature characterization (split cavity resonator with 2 thermal cycles). The orange line in the Fig. 10 shows the relative permittivity at 10 GHz of an Alumina substrate of thickness 0.376mm obtained by the traditional technique.

At 75°C, the permittivity of Alumina obtained by the traditional technique is 9.58. The red curve in the Fig. 10 shows the permittivity of Alumina sample obtained by the new technique. The results show a good agreement between the two techniques. The differences in values are negligible.

The orange line in the Fig. 11 shows the loss tangent at 10 GHz of the Alumina substrate obtained by the traditional technique.

At 75°C, the loss tangent of the Alumina obtained by the traditional technique is $1.9*10^{-4}$. The red curve in Fig. 11 shows the loss tangent of the Alumina sample obtained by the new technique. The offset of 1.3% in the loss tangent between the two techniques can be explained by the use of two different vector network analyzers with no calibration for the measurement of coupling to the one used in the traditional technique.

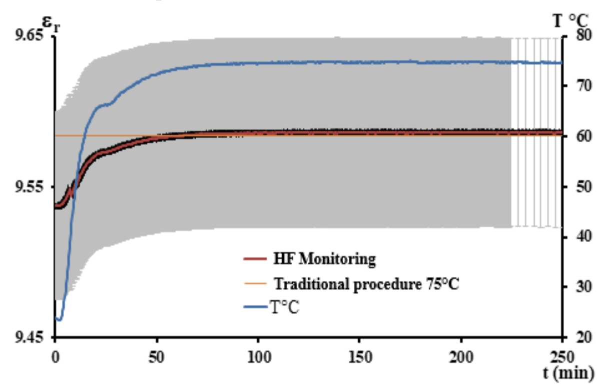

Fig. 10. Evolution of the permittivity in function of the thermal cycle.

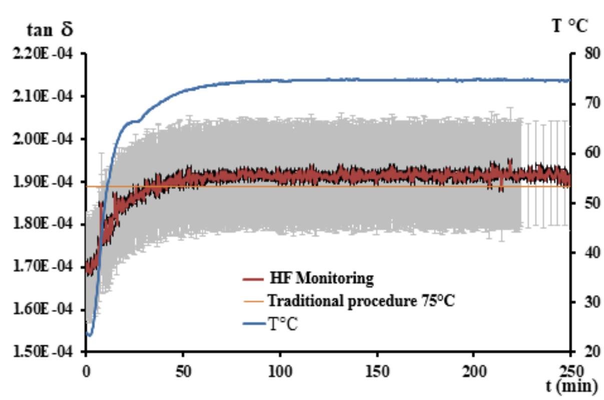

Fig.11. Evolution of loss tangent in function of thermal cycle.

By this method, we avoid the use of conductivity and dimensions expressions as functions of the temperature which are approximate models given the difficulty of the control of the temperature cartography of the cavity during the thermal cycle. The duration of the thermal cycles required for obtaining thermal equilibrium of the device is evaluated by the stabilization of measured resonance frequencies depending on the temperature in a range of ±50 kHz. With this criterion, the time required for this alumina is of the order of two hours.

5. Conclusion

Simultaneous temperature characterization of split cylinder resonator and dielectric samples during a single thermal cycle and in real time has been demonstrated. Electromagnetic simulations have confirmed the low disturbance caused by a material for the TE_{012} and TE_{014} modes. The conductivity measured by the TE_{012} mode, with a loaded cavity, is identical to that measured when the cavity is unloaded. Measurements of the cavity dimensions carried out at ambient temperature using the two couples show a small shift of the cavity radius (> 3µm). These small offsets lead, for example for the considered alumina, to a shift of about $\Delta\varepsilon'/\varepsilon' \approx 0{,}2\%$ and $\Delta\tan\delta/\tan\delta \approx 0{,}1\%$. A temperature measurement was performed for an unloaded cavity and a loaded one using the two couples to experimentally estimate the expansion of the cavity. Finally, the characterization of an alumina sample during a thermal cycle carried out using the new technique and the traditional one shows a good agreement for the permittivities and the loss tangents obtained by the two techniques.

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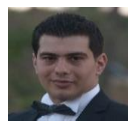
Biography of the authors



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