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QUICK CONTACT LESS THERMAL ANALYSE OF RUBBER BLENDS

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ABSTRACT
In the paper we present theoretical and experimental results of two variants contactless thermal analyses (CTA) for determination of specific heat capacity, thermal diffusivity, and thermal conductivity. Results obtained by this method were tested by independent contact method. All of the obtained results are in a very good coincidence.
Keywords: Contact less measurements, thermal properties, rubber blend

1. INTRODUCTION
The measurements of thermal conductivity, heat capacity and thermal diffusivity play an important role in rubber industry, mainly in tyre construction, because these values and their changes directly influence the instantaneous value of viscosity, loss factor tgδ and by means of that the adhesion of tyre to a road surface.
General theoretical aspects of heat transport in solid materials are described for example in the work [1]. Very effective contact less method for the study of thermal properties of materials is the infrared thermography which possibilities are widely analyzed in the work [2]. The temperature profile of a tyre was tested by scanning termovision camera in the work [3]. Interesting flash method was successfully tested for thermal properties of metal samples. Obtained results are presented in [4]. Nevertheless this method is not absolutely contact less. From the theoretical point of view we've applied presented theoretical results. Contact methods of thermo parameters measurement need in general relatively complicated electronic equipment. One of them was described in [5,6]. In present work we use this apparatus as a reference for determination of thermal conductivity λ [W/m.K] and diffusivity α [m^2/s].
In this work we present fully automatic and fully contact less method of heat capacity, thermal diffusivity, and thermal conductivity determination based on one measurement. Method is proper for measurement of thermal properties of materials with small thermal conductivity such as rubber blends, where it was applied.
For thermal conductivity λ [W/m.K] we can put down the well known relation

$$\alpha = \frac{\lambda}{\rho c} \qquad (1)$$

where ρ [kgm^{-3}] is the sample density and c[J $kg^{-1}K^{-1}$] is the specific heat capacity [2].
Two relations for determination of thermal diffusivity α through the sample of thickness L were derived [4]

$$\alpha = 1{,}38\, L^2 / \pi^2 t_{1/2} \qquad (2)$$

or

$$\alpha = 0{,}48\, L^2 / \pi^2 t_x \qquad (3)$$

where the sense of temperatures determined by both relations 5 and 6 is clear from Figure 1. L is the sample thickness. Relation between effective time and time corresponding to the maximum is given by relation

$$t^{ef}=1{,}6t_M \ . \qquad (4)$$

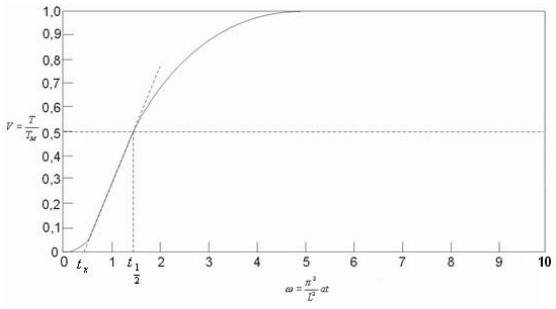

Figure 1. Theoretical plot in dimension less parameters for determination of characteristic times determined by equations 2 and 3 [4]

2. EXPERIMENTAL RESULTS AND DISCUSSION

At the beginning of discussion it is necessary to underline the fact, that the CTA in presented form was developed for measurement of thermal parameters of rubber and other materials with relatively small value of thermal conductivity. Presented measurements of metals are only supplementary in order to determine the absorbed heat Q.

In the first part of presented work (solution for relatively big samples) we have used polystyrene calorimeter, where the tested sample (Cu,Al, rubber blend) of rectangular shape with dimensions approximately equal to (0,09x0.11x0,0014)m was placed. The thickness of rubber blend sample is given later. Schema of the apparatus is in Figure 2. The sample was illuminated trough halogen lamp (electrical power1500W) switched on by computer. We used the pyro-sensor type Raytek THERMALERT MID 02 placed at the rear side near the surface of measured sample in order to sense the temperature. The whole measuring process was controlled and evaluated by special software which automatically switches the lamp on, measures the time-temperature dependence of pyrosensor response, and determines the temperature difference ΔT from measured data (see below). Every value was measured ten times in order to obtain repetition ability of the apparatus. Then the full set of values was transformed to software Matlab. After application of proper regression procedure of measured time-temperature dependences obtained from pyrosensor response by Matlab we obtained following values: t_M (according to equations 2 and 4), absorbed heat Q, specific heat capacity c, thermal diffusivity α and thermal conductivity λ.

We started the experimental analysis of results from measurement of heat adsorbed in the sample. We calculated it from calorimetric equation Q=mcΔT for Cu sample, with the table value of c_{Cu} = 383 J/kg.K.

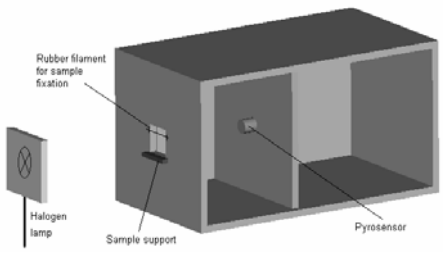

Figure 2. Lateral view on experimental setup

Surface of the Cu sample was covered on both sides of the sample by mat black spray. The heat absorbed by Cu sample was approximately equal Q_{Cu} = (61,81±0,02) J. We determined ΔT as a temperature difference of ambient temperature and maximum surface temperature measured by pyro-sensor on the rare sample surface. The validity of such experimental procedure was tested on Al sample of the same dimensions, also covered by the same mat black spray. In this case we calculated specific heat capacity of Al sample according to calorimetric equation at "known" absorbed heat Q_{Cu} determined on the base of previous experiment. Mean measured value of c_{Al} was (883,67±0,04) J/kg.K. Table value was 896J/kg.K what corresponds to difference of both values of c_{Al} approximately equal to 1, 4 %. So we can conclude that described measurement of Q gives the relevant values.

Later the rubber blend sample of rectangular shape, with the thickness of 0,002m was measured the same way. Geometry of the experiment and the value of active surface were the same in all experiments. For the evaluation of c_{rubb} we used the same absorbed heat determined for Cu sample Q_{Cu}. The surface of rubber was also covered by black spray on both sides of the sample, similarly as in previous cases. It is also necessary to use the black spray film on the surface of other, not black materials. Using the method described above we obtained mean value of c_{rubb} = (1514,80±30,31) J/kg.K.

Because we didn't have the reference table value for this blend we realized the reference measurement by analyzer Perkin Elmer, type Diamond DSC (differential scanning calorimeter). Mean value at 38°C was c_{rubb}=(1678±0,043) $Jkg^{-1}K^{-1}$, what corresponds to ten percent deviation of values obtained by both methods. It is necessary to underline that the other sample of the same composition has been used for DSC measurement as for CTA. The influence of local sample inhomogenity is possible, because the mass necessary for DSC measurement is approximately on the level of ten milligrams.

In the next step we will judge the results of α and λ measurements on rubber blend sample obtained by CTA and independent contact method used in work [5,6].

Now we can compare results obtained by both methods. The mean value of both α and λ obtained from CTA were α=(1,81±0,03).10-7 m^2s and λ=(0,344±0,004) W/mK. Sample density was ρ=(0,99973±0,00006).103kg/m^3. The rubber thickness was $2{,}2.10^{-3}$m, other dimensions were the same as for metal samples. The results obtained by apparatus described in works [5,6] for both values are α=(1,85±0,02).$10^{-7}m^2s$ and λ=(0,31105±0,0004) W/mK. Specific thermal heat capacity calculated from these measurements according to relation 1 is equal to c=1691,8 $Jkg^{-1}K^{-1}$ what represents deviation from CTA value equal to approximately twelve percent. The sample specific density calculated from relation 1 equals ρ = 993.66 kgm^{-3} what is in excellent agreement with value reported above. Difference between diffusivities α obtained from both experiments is approximately equal to two percent. Difference between both values of thermal conductivity λ is on the level of ten percent. From presented results obtained for c, λ and α it is clearly seen that shown method gives the reproducible results with the good repetition ability. The obtained results are in a very good coincidence with other independent methods for measurement of c, α and λ.

In the next part of our contribution (contact less solution for relatively small samples) we have used compact measuring, fully automatic system which is presented in the Figure 3 [details are in www.fpt.tnuni.sk/umtv (our products)]. The tested samples were Cd (as the reference) and rubber blend, all of cylindrical shape with dimensions Ø= 12mm and thickness approximately 2mm .The sample was illuminated trough halogen lamp (electrical power 200W) switched on by computer. We used the pyro-sensor type Raytek THERMALERT MID 02 placed at the rear side near the surface of measured sample in order to sense the temperature. The whole measuring process as in previous case was controlled and evaluated by special software which automatically switches the lamp on, measures the time-temperature dependence of pyrosensor response, determines the temperature difference ΔT from measured data (see above). Every value was measured ten times in order to obtain repetition ability of the apparatus.

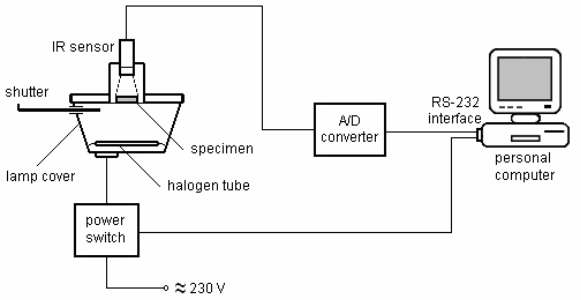

Figure 3. Schema of the thermal analyzer

Cd sample was covered on both sides of the sample by mat black spray. The measured value of c_{Cd} was (225,33±1,6) J/kg.K and corresponding table value is equal 231 J/kg.K. Difference both values is approximately 2,5 %. So we can conclude that described measurement of Q gives the relevant values. Tested rubber blends of a diameter 12mm (covered by mat black spray on both sides) and the same composition as in above described experiments offer a very close values of measured thermal parameters $\alpha=(1{,}91\pm0{,}02).10^{-7}$ m^2s and $\lambda=(0{,}37\pm0{,}006)$ W/mK. Differences both presented variants so do not exceed ten percent.

3. CONCLUSIONS

Presented CTA (in both versions) is fully contact less, fully automatic and it is proper for testing of materials with relatively small thermal conductivity. From one measurement it is possible to determine specific heat capacity, thermal conductivity and thermal diffusivity with relatively high precision and very good reproducibility. Absorbed heat Q and density are input parameters. In both presented version it is possible to measure large dimension scale of samples.

4. REFERENCES

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