THEORETICAL ANALYSIS OF CARBON FELT COMPRESSING INFLUENCE ON ELECTRICAL CONDUCTIVITY IN DIVERSE ENVIRONMENTS

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ABSTRACT

Electro-conductive carbon felt (CF) material owing good electrical and hydrodynamic properties is found in numerous electrochemical applications for electrodes design in redox flow batteries, fuel cells, electrochemical desalination apparatus etc. Its structure is composed by bonded together having different lengths carbon filaments which create porous structure with significant internal surface for ensuring rapid electrochemical redox reactions.

CF electro-conductivity is ensured mostly by long bonded carbon fibers located between electrodes and connecting at the same time both of electrodes surfaces. Electro-conductivity property of CF is extremely important for the electrochemical appliances and should be increased as much as possible. It is known that increased electrical conductivity can be achieved by a small pressing of CF to the direction of conducting electrodes substrate. Increasing pressing on a felt diminishes its volume thus providing supplementary electrical contacts of carbon filaments inside a felt and between electrodes. Such circumstance causing improved electrical conductivity and therefore is desirable to some extent. Moreover, multiplying internal interconnects enhance an active surface of a felt electrode boosting redox chemical reactions. However, additional CF pressing diminishes its hydrodynamic and aerodynamic permeability and this fact should be considered for the optimal design of electrochemical cells.

CF electrodes are used in different environmental conditions. For heating elements, it is used as a dry substance in an air or vacuum. Yet, in electrochemical cells, CF electrodes are immersed in the electrolyte, which is capable to change fibers interconnections and influence conductivity. Consequently, the impact of electrolyte characteristics on CF electro-conductivity should be investigated as well.

Multiple researchers describe CF resistivity by experimental empirical approaches having as results math functions characterizing the behavior of its electroconductivity. However, there is a lack of theoretical models able to connect CF structure properties with its electrical parameters.

The strictly theoretical models are particularly important for improving future frameworks of CF as well as for designers of specific electrochemical systems having to know the electrical characteristics of are being developed cells.

The presented article provides results of CF conductivity measurements in different environmental conditions and the describing CF electric properties theoretical model based on a stochastic method.

The article includes: Introduction, Methodology of Experimental Research, Conductivity Measurements, Math Interpretation of Experimental Data, Theoretical Model of Electrical Conductivity and Conclusions.

Keywords: Carbon felt, electrical conductivity, dielectric liquids.

INTRODUCTION

CF material is widely used for electrodes substances in different applications including electrochemical [1-7]. For today the most prominent utilization of CF is in the Vanadium Redox and others flow batteries regards its good electrical conductivity, hydraulic permeability and durability to sustain in strong acid and alkali electrolytes [8-20].

The following research works [21-30] dedicated for investigation and measurements of different including electrical properties of CF. Factual physical parameters of rayon-based CF and which is being manufactured in Mersen C^{o}. as large sheets with a width of several mm up to a few cm could be found in [31].

As a material for heating elements or for electrodes, carbon felt must possess necessary electric conductivity. Its conductivity could be improved by felt squeezing because of diminishing distance between carbon fibers and producing additional quantities of fibers interconnections. However, this automatically causes a decrease of an empty volume and cross-section of microchannels connecting pores inside felt framework. The results of this process it a decrease of a felt permeability or increase of hydraulic resistance preventing free electrolyte flow through electrode.

Since for optimal development of a specific appliance with CF one must consider electrical properties, numerous scientific works studied they as a rule experimentally with the following analysis of obtained results. More valuable for designers of future CF may be theoretical description of the conductivity. Considering a stochastic CF framework nature theoretical model can be developed on a stochastic approach only.

One of the first measurements of CF electrical resistivity was carried out for furnace heating elements [3]. The work [26] describes the design of a new bipolar cell with proton-exchange-membrane where flat electrodes were made of flexible and loose carbon fiber bunches representing an analog of CF. Due to the need to ensure good electrochemical reactions on electrodes, tests of CF resistivity were conducted. For the first time, experimental results of CF area-specific resistance (ASR) were represented as a function of a squeezing pressure. It should be noted that ASR represents a parameter equal to a total resistance in the perpendicular-plane direction of CF electrodes divided by the value of its area. The decrease of the bulk resistance was observed as an exponential function with negative exponent value depending on the pressure applied to the CF electrode.

Important and original results of measured ASR for CF [27] were represented as a function of a compression force. The exponential diminishing of a resistance versus compression pressure (as in [26]) was observed. However, different coefficients of the exponential function were estimated, which depend on the type of construction and adhesive materials.

The above-mentioned works don't relate a porosity to CF electric conductivity and describe this parameter integratively. Nevertheless, the question of interest is analytical expressions describing resistance as a function of CF porosity and a specific resistivity of a carbon filaments. Such formulations were obtained and shown in [28-30]. Above-pointed specific area electrical resistance which is denominated as (ρ_{felt}) [28] is described by electrical resistivity of a carbon filaments material (ρ_{carbon}) and felt porosity:

$$\rho_{felt} = \frac{4}{(1-\lambda)}\rho_{carbon} \quad , \qquad (1)$$

Expression (1) is in good agreement with the measurements results provided in [29]. ASR of CF electrodes (denoted as (R_{ASR}) in [30]) for vanadium redox batteries was determined experimentally. Approximation of R_{ASR} [$m\Omega\cdot cm^2$] versus V_f (fiber volume fraction, %, the opposite of the porosity λ, i.e. $V_f/100=1-\lambda$) was evaluated by following exponential function:

$$R_{ASR} = 144.456 \times \left(3-\frac{10}{V_f}\right)^{-1.117} \quad , \qquad (2)$$

Approximation (2) that was obtained for specific vanadium redox batteries is more accurate than that of (1). But it doesn't consider different filament materials and is applicable only for the pure carbon fibers. Furthermore, all experimental measurements were carried out in dry conditions only, i.e. in air. Owing to this obstacle, the expression (2) can’t precisely predict electrical conductivity of a CF being immersed in different electrolytes. However, real electrodes work in electrochemical cells filled with different electrolytes and our experiments showed the dependence of resistivity on the type of electrolyte. Resistivity or conductance of CF seems to be influenced by some liquids 'specific properties conducive to the penetration of fluid between fibers or between fibers and electrodes and in this way decreasing the connections density and diminishing the felt conductivity or conversely causing the decrease of resistance.

Theoretical modeling of CF conductivity owing to a significantly stochastic framework of felt structure, may be developed by a stochastic approach with some reasonable assumptions and this way seems to be the most valuable. Applicable methods for this aim are represented in [31-33].

Therefore, it was decided to conduct a parallel study of both electrical and hydraulic properties of the same CF. The results of the experimental research together with the analytical approximations are presented below.

II. Methodology of Experimental Research

The experimental research main objective was the investigation and determination of electrical properties of CF in a wide range of external conditions.

Characteristics of CF

The CF for experiments was a rayon-based material made from high-quality carbon fibers. It has a porous structure with a large superficial surface. Rayon based CF from the well-known producer "Mersen" USA Greenville-MI Corp [26] was the matter for the tests. Its structure shown in (Fig.1). Additional characteristics of this kind of CF could be found in Table (from [34]) .

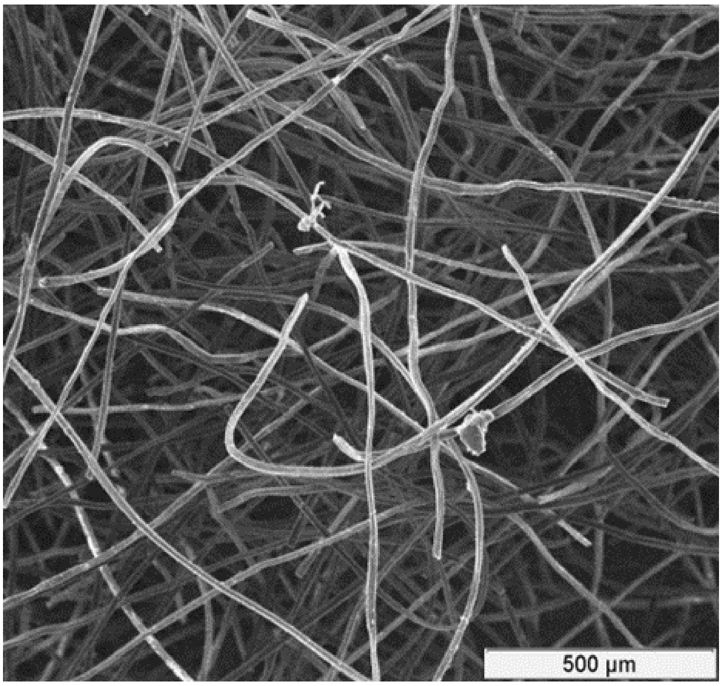


Fig.1. A magnified picture of CF structure

Table 1. Mechanical properties of a CF

	Average	Standard deviation
Fiber diameter, μm	19.2	1.66
Felt density, [kg/m^3], ρ_f	88	-
Carbon density, [kg/m^3], ρ_c	1954	-
Porosity, [%], θ	95.5	-
Relative carbon volume, p.u. [%], V	0.045 (4.5%)	-
Specific felt surface, [m^{-1}], S	$9.8 \cdot 10^3$	-

The special testing cell was made (Fig.2), which construction permits electric conductivity measurements for differently squeezed CF. Cells dimensions (Fig.3) allowed testing felt samples 47×28.7 mm whereas felts height has been changed by pressing from 6.6 to 1.2 mm by four screw bolts. A symmetry of a felt squeezing and its height measurements have been provided by electronic caliber having 0.01 mm accuracy.

Electrical conductivity was measured for the felt is in the cell between two thin copper sheets simulating electrode's substrate.

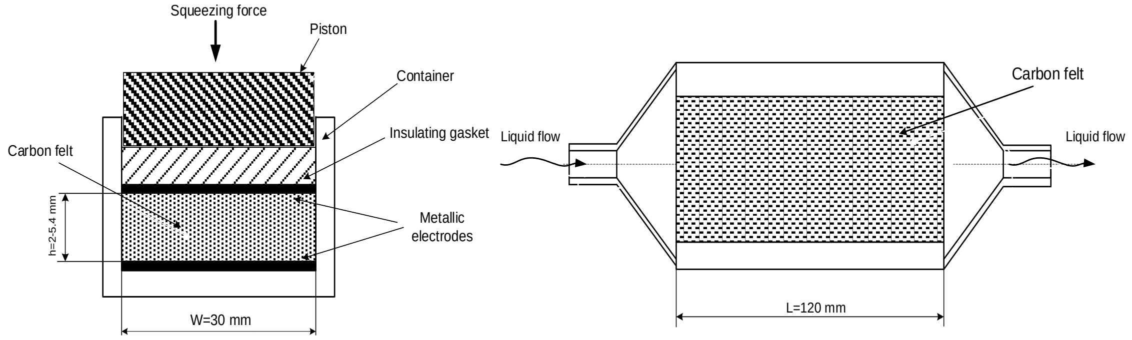


Fig.3. Special experimental cell for conductance measurements

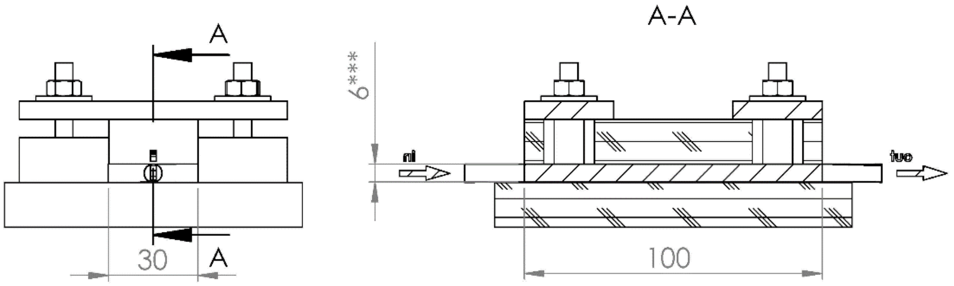


Fig.3. Dimensions of the experimental cell (from [21])

Electrical conductivity was determined in the cell for differently squeezed CFs, firstly, in dry conditions and later when CF was immersed into three different dielectric liquids having dissimilar electrical permittivity. These liquids were glycerol, alcohol, and cyclohexane. Conductivity estimation was provided by bench type R/L/C METER IX 3131B able to measure resistance by applying 100Hz, 120 Hz, 1kHz, and 10 kHz AC frequencies with the error of

~0.6%. In addition, resistance by applying DC was measured by Fluke multimeter. Results of conductivity measurements are presented in the following sections.

III. Conductivity Measurements

Electrical measurements outcomes will be presented here both for dry felt electrodes and for those immersed in different however dielectric (nonconductive) liquids. The reason for applying dielectric liquids was preventing the influences of own (ion) liquid conductivity on the resistance of the felt which was the main object of the investigation. Major supposition for the conductivity description was an assumption that the resistance of the felt determined mainly by the density of filament's interconnections inside a felt as well as by the density of filament's contacts with electrode's surfaces.

Three dissimilar liquids were chosen: glycerol, alcohol, and cyclohexane all with significantly different electric permittivity ε. Below is the Table 2 representing the most important physical parameters of them. Resistance values of dry CF are shown in Table 3.

Table 2. Physical parameters of glycerol, alcohol, and cyclohexane.

Parameter	Liquid		
	Glycerol	Alcohol	Cyclohexane
Density, [g/cm^3] (25°C)	1.26	0.789	0.8
Dielectric constant, ε_r, [p.u.], (0.57MHz, 25°C)	~42.5	~21.6	~2.02
Electrical conductivity, [$(\Omega\cdot\text{cm})^{-1}$], 25°C	$5\cdot10^{-8}$	$\sim1\cdot10^{-6}$	$<5\cdot10^{-9}$
Viscosity, [Pa·s], 20°C (30°C)	141 (61.2)	~0.11	0.61

Table 3. Resistance of dry CF.

Parameter		h, [mm]						
		6.2	6.1	4.9	4.45	3.7	1.55	1.1
		Volume decrease, [%]						
		0	2	21	28	40	74	81
Rdc [mΩ/cm^2]		48.184	41.2	18.2	10.100	6.672	5.189	5.930
Rac, [mΩ/cm^2]	100, [Hz]	54.855	39.8	16.3	6.820	5.930	4.374	4.374
	120, [Hz]	54.633	38.9	16.6	7.042	6.079	4.522	4.299
	1, [kHz]	54.633	40.1	15.9	6.894	6.375	4.522	4.225
	10, [kHz]	49.296	38.7	14.9	6.746	5.930	4.670	4.299
Rac average, [mΩ/cm^2]		53.354	39.740	16.38	7.520	6.197	4.655	4.626

Measurements approve the decrease of CF resistivity during it pressing in the direction perpendicular to its surface. In the beginning, CF resistance falls fast and in the continuation, resistance decrease becomes slower. The more inclusive analysis is given in the following section.

IV. Math Interpretation of Experimental Data

We suggested representing electrical CF properties resistance by an opposite parameter - average surface conductance (ASC) instead of the use of ASR. The advantages of such representation will be seen later.

A typical graph of ASC versus felt squeezing D, [%], is shown in Fig.4.

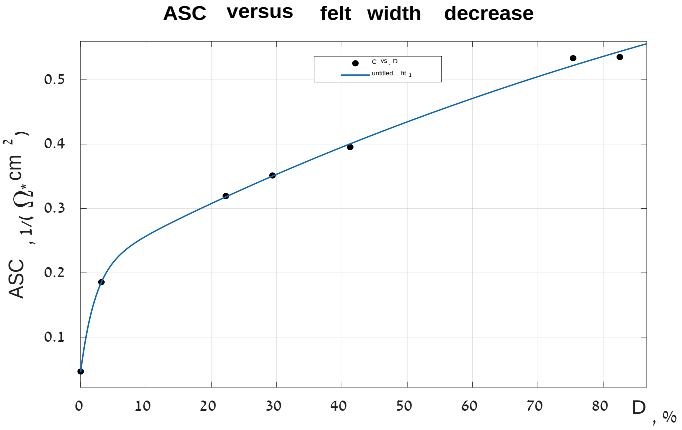


Fig.4. ASC of a dry CF versus relative felt decrease.

Similar graphs were obtained for CF in glycerol, alcohol, and cyclohexane. The behavior of ASC $[\frac{1}{\Omega\cdot cm^2}]$ can be sufficiently described by the exponential function (3) precisely approximating virtual data:

$$ASC = a\cdot\left(1-e^{-b\cdot D}\right)+c\,, \qquad (3)$$

Below is given a Table 4 representing magnitudes of the coefficients a, b and c for describing ASC in different environments. The proximity of the theoretical function to real data is estimated by coefficient R^2 having significantly high value (see Table 4).

Table 4. Coefficients quantities of approximating function for ASC

ε_r	**ASC**	**ASC=a*(1-exp(-b·D))+c**			**coeff. R^2**
		a	**b**	**c**	
1	**dry**	**0.5071**	**0.02488**	**0.0933**	**0.969**
42.5	**glycerol**	**0.9134**	**0.01278**	**0.0996**	**0.967**
21.6	**alcohol**	**0.4269**	**0.02377**	**0.322**	**0.982**
2.02	**cyclohexane**	**0.5024**	**0.02008**	**0.2116**	**0.988**

One can put the mind on the dependence of ASC from liquid dielectric parameters. Obviously, enlarged electric permittivity ε positively influences CF conductivity. It could be explained by the increasing the density on internal filaments interconnections between themselves and electrodes surfaces. Owing to the interconnections between carbon filament as the main factor, its relative increase $(K_{inc})_{rel}$ was calculated as:

$$\left(K_{inc}\right)_{rel} = \frac{\left(ASC\right)_{Di}}{\left(ASC\right)_{D_0}}\cdot\frac{\left(V_0\right)}{\left(V_i\right)}, \qquad (4)$$

Where: $(ASC)_{Di}$ and $(ASC)_{D0}$ conductance for current CF width squeezing versus its initial value; V_0 and V_i initial volume and its current values.

R_{incr} has a common approximating expression (4) and a similar graphic interpretation (Fig.5).

$$\left(K_{inc}\right)_{rel} = A\cdot(e^{B\cdot D}-1)+1\,, \qquad (5)$$

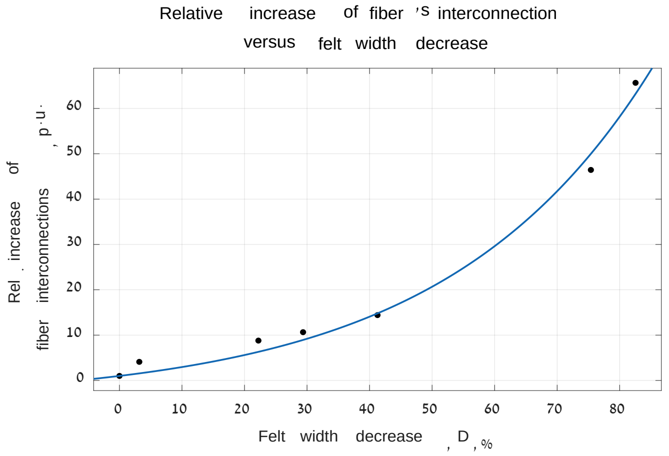


Fig.5. The relative increase of filaments interconnections versus relative volume decrease

Considering the measurements results and coefficients of approximation function (5) for each liquid, Table 5 that presenting quantities of R_{incr} versus CF width decrease, was established.

Table 5. Coefficients quantities of approximating function for the relative increase of filaments interconnections

ε_r	R_{incr}	$R_{incr}=A(e^{B\cdot D}-1)+1$		coeff. R^2
		A	B	
1	dry	5.499	0.03042	0.991
42.5	glycerol	2.832	0.0322	0.98
21.6	alcohol	0.5523	0.05052	0.996
2.02	cyclohexane	0.8402	0.03486	0.986

V. Theoretical Model of Electrical Conductivity

Experimental results prove the pressing as a major factor in increasing CF conductivity. Moderate influence on a conductivity as well has a dielectric constant of a liquid whereat CF is submerged in. Intuitively it is understandable that CF pressing causes increasing interconnections between carbon filaments and electrodes surfaces. As a result, more conductive ways for electrons passing from one electrode to another is created. Therefore, electrical conductivity is growing up and resistance is diminishing down. The increase of interconnections obviously has a saturation effect since any of a CF volume has a restricted number of fibers able to conduct electricity. Therefore, one can observe a satiating rise of CF conductivity during a volume decrease. The similar impact on fibers interconnections and therefore, on CF conductivity may have liquids dielectric constant. Electrolytes having different dielectric constants can promote or prevent additional conjunctions between fibers and electrodes thus changing the resistivity of a CF. Bearing in mind relatively low influence of dielectric electrolyte properties on CF conductivity it was decided to create an analytical model of a dry felt and to correct its results by a coefficient that considers specific electrolyte (as it was represented in Section IV).

The analytical model should connect an increasing conductivity with CF compressing. The statistical method was chosen for the purpose. It describes a community of internal carbon fibers as an array of stochastically dispersed filaments each of which has a different length, and angular location in the internal CF volume. Owing to the assumption of the filament diameters equality (that is supported by diameters measurements, see Table 1), it is possible to describe a resistance (conductivity) of each filament proportional to its length. Only those of filaments, which contact both of electrodes, provide real CF conductivity. Its value is summarized from the individual conductivities of particular fibers. Pressing of CF causes the increase of fibers number, which simultaneously connecting two electrodes. Knowing the dispersion of individual lengths (resistances) and a scattering of their angular locations statistics may provide the calculation of a summarized conductivity for each CF height. Therefore, this will allow the estimation of CF conductivity as a function of its relative squeezing.

On the first step the frequency (probability) of filaments lengths was measured by optical method and described by Gamma-Function (6). It was assumed for the simplicity that filament has the same diameter and is represented by a line segment. Therefore, each resistance is strictly proportional to segments length whereas the conductivity is inversely proportional to it. Knowing filament length provides the estimation of its conductivity as in (7):

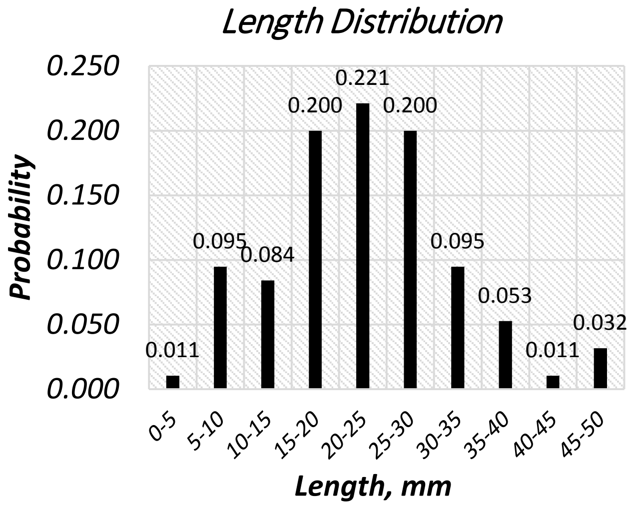


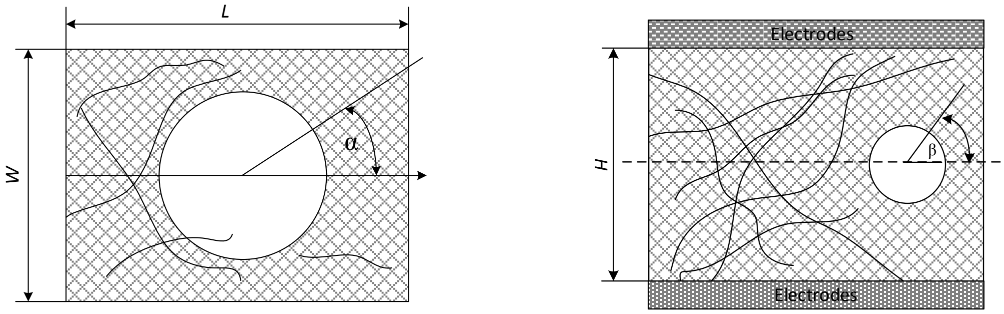
Fig.6. The frequency (probability) of filaments lengths.

$$f(L;\delta,\gamma)=\frac{\beta^{\delta}L^{\delta-1}e^{-\gamma x}}{\Gamma(\delta)} \quad \text{for } L>0 \text{ and } \delta,\gamma>0, \qquad (6)$$

Where: δ and γ are gamma-function coefficients.

$$G(L)=\frac{\pi d^2/4}{\rho_C\cdot L},\quad 1/\Omega, \qquad (7)$$

For creating a stochastic model of conductivity were considered filaments lengths, horizontally and vertically distributed angles: α and β (Fig.7, a, b)



a) b)

Fig.7. Angles (α and β) distributions inside CF space.

Probabilities of horizontal and vertical angles that were estimated as well by optical method and are shown in Fig. 8 (a, b).

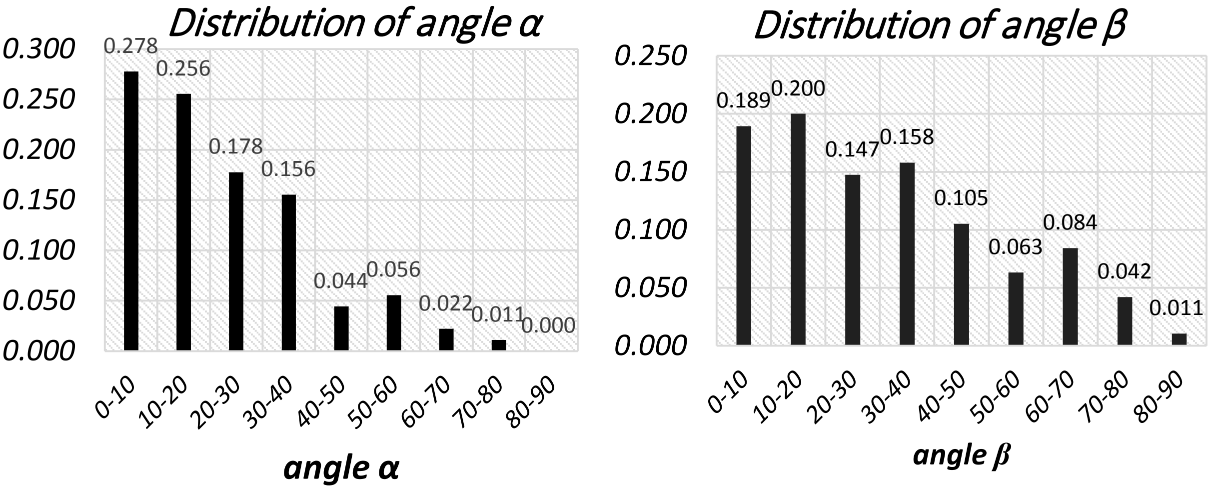


Fig.8. Probabilities of horizontal and vertical angles

It is required to summarize the conductance of all filament which are touching both of electrodes at the same time. For this purpose, the height h (Fig.8) must be assessed. Those of filaments which height h is equal or bigger than the total height of a felt H are participating with their conductance in the current flow between electrodes. The height h can be calculated through a length of a filament (segment OA) and an angle φ:

$$h = L\sin(\varphi), \qquad (8)$$

In turn, angle φ:

$$\cos\varphi = \frac{\cos\beta}{\sqrt{1-\sin^2\alpha\cdot\sin^2\beta}} \Rightarrow \varphi = \arccos\left(\frac{\cos\beta}{\sqrt{1-\sin^2\alpha\cdot\sin^2\beta}}\right), \qquad (9)$$

Below is located Fig.8 with the axis XYZ. Vector Z denotes vertical coordinate determining a distance between electrodes surfaces, vectors X and Y determine the coordinate width and length.

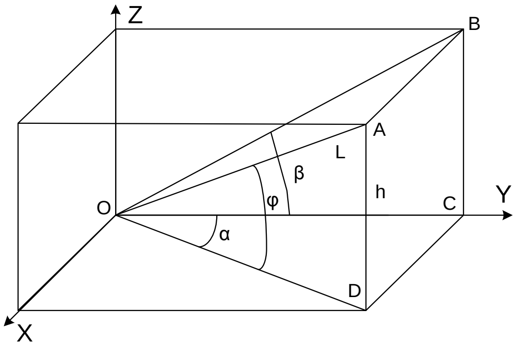
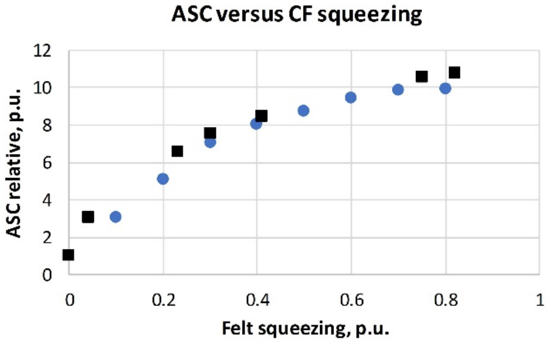


Fig.8. The position of angles (α, β and φ) for individual filaments inside a felt space.

Total conductivity of a CF squeezed to the height H is assessed by summarizing conductivities of all filaments which at the same time touching both electrodes. That is, it requires to consider all of them which h coordinate is bigger than the height of a felt plate. Representing a community of all filaments as a continuous countless set, the conductivity may be calculated as a complex integral:

$$G\left(h\right)=K_G L\int_H^{\infty}\left(\int_{\arcsin\left(\frac{h}{l}\right)}^{\pi/2} p_\beta d\varphi\right)p_l\frac{dl}{l}, \qquad (10)$$

The results of solution of equation (10) that was carried out by special MATLAB procedure are graphically represented in Fig.9. Experimental data are denoted by black squares.



A comparison between real ASC data to theoretical prediction verifies sufficient exactness and equity developed method for CF conductance estimation.

CONCLUSIONS

1. CF material has widespread requests in different appliances including heating elements and diverse electrochemical cells.
2. Electrical (electronic) conductivity of CF plays a crucial role for electrons to reach the surface of the electrodes and to participate in reactions. Likewise, electrical resistivity (the inverse of electrical conductivity) plays a significant role in generating heat or causing electrical losses diminishing the efficiency of electrochemical equipment.
3. Owing to a significant assignment of CF resistivity and conductivity, a special comprehensive investigation was carried out aiming to find magnitudes of these parameters and to develop analytical model for describing electrical properties.
4. Electronic resistivity was measured by a special device applying for this purpose DC and AC currents with different (100 Hz, 120 Hz, 1 kHz and 10 kHz) frequencies. Considering the requirement to verify the influence of dielectric liquid parameters on CF conductivity, four sets of tests were performed. Resistance was measured in dry conditions and with the CF immersed in different (glycerol, alcohol, and cyclohexane) non-conducting (dielectric) liquids. Usage of dielectric liquids instead of real electrolytes was justified to prevent an influence of electrolyte ionic conductivity on the measurement results.
5. It was observed that electrical resistivity was diminished during felt compression having a non-linear relation versus volume decrease like the negative exponential function. In the initial stage of volume decrease, the resistance drops quickly. However, after 80-60% of its initial value, additional compression has a negligible effect on conductivity.
6. Electrical conductivity moderately depends on liquid permittivity properties. Dielectric constant (ε), among other liquid parameters, obviously has a main role influencing the quantity of interconnection between carbon filaments and electrodes. The increase of ε causes a slight improvement in CF conductivity.
7. Analytical model of CF conductivity was developed and verified. It is based on stochastic representation of filaments parameters describing their lengths and angular position inside CF framework.

8. Applied gamma-functions which determines probability of filaments lengths distribution and their stochastic angular dispersion in the internal space allowed analytically approximate dependence of conductivity versus CF squeezing.
9. Obtained results prove developed method allowing with enough exactness describe the influences of framework stochastic properties on CF electrical conductivity.
10. Should be important to continue this investigation for deep learning of the impact that produce angular filament dispersion on a conductivity as well as its dependence from filament lengths.

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