Hardware and software implementation of a parallel-plates rotational rheometer

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***Abstract*—A rotational strain controlled rheometer with parallel-plate was designed and realized in SMT configuration, which allows exhaustively to characterize materials having highly viscoelastic properties. In this particular case, composite materials, reinforced by dispersed particles, were tested: they are constituted by the elastomer polydimethylsiloxane (PDMS), with addition of different filler concentrations, in particular carbon black, electrically conductive elastomers useful in electronic devices.The study of rheological material properties can contribute to the better realization and manufacturing process of the product. Since the dynamic properties evaluation has been done through oscillatory testing at fixed range of frequencies, in order to measure the complex modulus G* of the material.**

***Index Terms*—Rheometer, Complex modulus G*, Viscoelastic, Carbon black**

I. Introduction

The linear viscoelasticity theory is a prerequisite for the understanding of the rheological characteristic of many materials [1]. Defining a viscoelastic system by properties intermediate between solid and a fluid. A dissipation of internal energy and hence the stress - deformation deviation usually are verified in two phases interaction of solid - liquid and elastic - viscose, respect to a perfectly elastic state. In fact, a perfect elastic response requires a potential energy acquired during deformation phase,restoring it totally in order to observe the transformation reversibility [2], [3], [4].

The major or lesser influence of the solid matrix on liquid phase causes an infinite variety of viscoelastic responses, whose extreme limits are the solid pure elastic and viscous fluid. Afterwards the main viscoelastic mathematical relations are explained widely in rheological analysis. In the physical phenomena, the real materials are identified with a continuous homogeneous and isotropic model [5].

The study of the rheological properties and viscosity measurement arise for exploration of materials dynamic behavior; a special equipment called *rheometers* are used to this purpose, which generally allow to measure dynamics (forces and torques) and kinematics (displacement, velocity and time) variables [6], [7]. Rheological quantity is obtained by mathematical equation solutions and experimental test, considering also some geometric parameters of the used instrument and physical parameters of tested material.

For instance, the polymeric materials are characterized by complex rheological properties, in particular for flow conditions in real applications. For this reason in the laboratory are created simple kinematically flow situations and easily controllable.

Operational protocols definition and processing of experimental results are fondamental for execution of rheological tests. Usually the rheological properties of polymers such as elastic, viscous and viscoelastic are typically determined using equipment operating in sweep frequencies, including the *DSR Dyanamic Shear Rheometer*. An independent variable, called forcing, varies over the time, and in general is considered a stress, deformation or deformation gradient.

II. Hardware Implementation

The strain controlled rotational rheometer are constituted by two parallel plates, the bottom plate is moved by a stepper-motor, which allows to apply a predetermined rotation speed, which thus determines a sliding flow inside the fluid interposed between the two plates. This motor can apply a torque in both directions, generating the oscillatory stresses; in this case the device can control both the intensity of the deformation that its frequency [8], [9].

The phase shift between the deformation and stress is determined by the transducer to measure the torque keeping the plate in motion, obtaining the stress undergone by fluid.

The measurement of viscoelastic properties have been analized at different frequencies using the rotational rheometer made in laboratory.

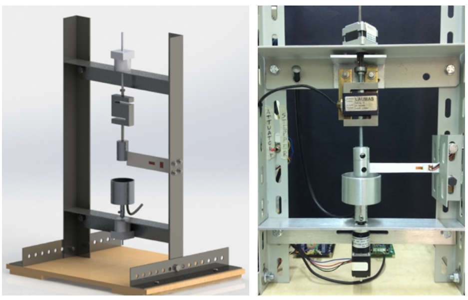

Fig. 1. 3D CAD model Solidworks of rheometer and its realization

The undercarriage of the testing machine is structured on one central axis, constituted by beams made of metal, with the L shape , fixed on a wooden base, which had been carried out to integrate to the test bench so as in order to reduce vibrations and oscillations during the testing.

The lower part of the rheometer machine is composed of a component, attributable to the cup shape, made up of a bushing for the connection with the stepper motor shaft, fixed by a screw. The upper part of the cup acts as a container in which it is located the specimen to be tested, and used for biological materials it allows to contain their physiological solution. This component was derivated by aluminum cylinder in the Heavy Equipment Mechanics Laboratory of the University of Catania, realized with the lathe and milling machine, dimensioning the component taking in account the available space presents between the two horizontal rails.

A motor *Phidgets 3321-0 - 28STH32 NEMA-11 Bipolar Stepper*, with integrated planetary gearbox Gearbox 27: 1 and stepper function, characterized by a maximum speed of 120 rpm and a rated torque of 1.4 Nm has been implemented [10], [11]. All system was designed as sine wave generator to apply the desired shear strain to the specimen.

Aluminum was chosen as the optimal solution to solve the problems, such as the excessive weight of the cup that could stress the lower stepper, and the oxidation caused by the saline solution for in-vitro tests on biological materials. Moreover, it has been provided the realization of a hole in the container, threaded to be able to connect a flexible rubber tube (Fig. 2).

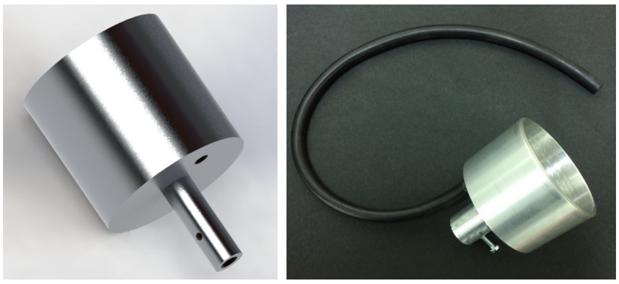

Fig. 2. 3D CAD model Solidworks of lower disk and its realization

An another stepper motor, on upper part of rheometer, equipped with a worm screw in order to convert its rotary motion into linear, and thus assume the function of the linear actuator to apply an appropriate compression to the specimen.

A load cell was tied to actuator, with a capacity of 150N, necessary for the detection of the compression force. In addition, the appropriate metal guides have been fixed to the upper beam, by bolts, in order to maintain in axis the load cell during the ascent and descent of the upper plate. The load cell is connected to a signal amplifier of Transducer Tecniques TMO-01 able to modify the voltage output of the load cell [mV], supplied with the correct voltage (12 VDC) by generator.

The other plate made of aluminum has the shape of a cylindrical punch. In its upper side the bearing housing has been obtained with mechanical interference. Using a bolt, inside of all system, enables to make it integral with the load cell and free rotation of upper plate in function of the viscoelastic response. The end of the punch in contact with the specimen has been knurled, in order to ensure a greater adherence and avoid slippage of its during the test (Fig. 3).

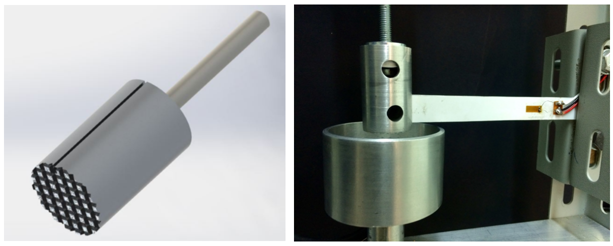

Fig. 3. 3D CAD model Solidworks of upper disk and sheet of styrene

By means a transducer, the phase difference is calculated between the angular deformation and the tangential stress. In this regard, specifically another type of load cell was made to measure the deformations undergone by the specimen during the test. In this case, the tested specimen can be compared to a band pass filter, having a similar frequency response. This load cell consists of a flexible styrene sheet (*Plasticard* with 1 mm thickness), containing a strain gauge configured in Wheatstone quarter-bridge, placed on the middle line of the foil to provide a measure without mistakes.

A lateral slot has been formed on the top plate, which allows the insertion of the foil, and the other side is stuck to the undercarriage by corner plates and the bolts.

The two motors have the same electrical characteristics, for this reason a single control device of Phidgets Controllers 1067 was chosen, with a sensitivity of 1/16 step, required for precise positioning, using a switch of activation of stepper-motor.

For the acquisition of the signals supplied by the load cell and strain gauge, a PXI platform of the National Instrument is used. It is able to offer solutions for high - performance measurement and automation systems. But the most important thing is the ability to perfectly synchronize the signals from the various sensors. In this project, the system implemented is as follows:

- Chassis NI PXIe - 1073
- NI PXIe - 6341 PXI Multifunction I/O Module for DAQ
- NI PXIe - 6341 PXI Multifunction I/O Module for DAQ
- Front-Mounting Terminal Block NI TB -4330 8Ch Bridge Input, necessary for the acquisition of the strain gauge.

The signal of the load cell is acquired by NI SCB - 68A and subsequently processed in the control system. Furthermore, all the above mentioned modules have a double insulation between each channel and the ground, for safety purposes and barrier against any noises during the testing.

III. Software Implementation

The rheometer is controlled by the CS control software developed in environment $NI\,LabVIEW^{tm}$, to activate the system and to analyze the results. The block diagram contained all the codes for the machine automation and algorithms to perform the required measures. To facilitate the debugging, the entire block diagram is divided into several sections, each designed to perform a specific function.

The first step of the measure process consists to apply compression of the sample until reaching the desired value, by driving the actuator. This compression is expressed by the percentage of the sample thickness, and already the upper plate touches the sample, the percentage of fixed displacement is applied.

The actuator is moved with a gradual descent up until to touch the sample, checking that the signal of the load cell is around zero, displaying the graph on *front panel*. When the top plate starts to compress the sample, a slight increase of the signal is verified. The value represented the regulation of the actuator movement depending on the desired compression rate is called *threshold*, in this is set at - 0.016. In this way the contact position is defined with extreme precision. The control logic of this mechanism is controlled by the While loop shown in Fig. 4. Within this loop a comparison between the signal of the load cell and the above-mentioned threshold value has been done, which is managed by the Flip - Flop SR custom-made in LabVIEW:

- If the signal of the load cell is below the threshold, the actuator will drop quickly with a continuous movement
- If this threshold value is exceeded, the actuator has touched the sample, resulting in a slowing it down, performing a controlled displacement and applying the percentage of compression.

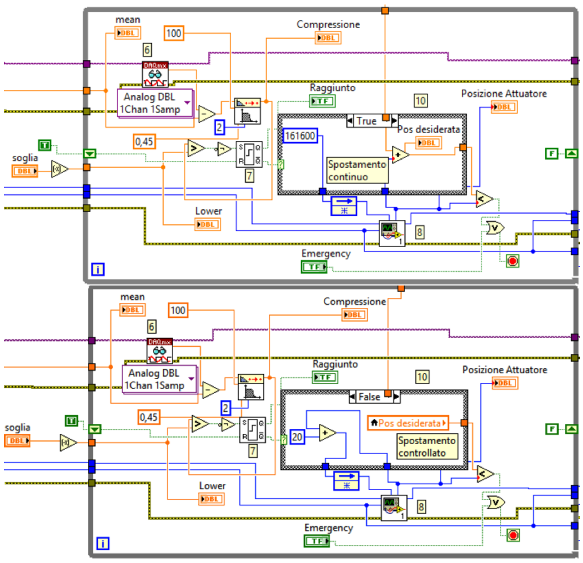

Fig. 4. Flip - Flop SR custom-made in $LabVIEW^{tm}$

The particular materials, as biological cartilage, require a certain waiting time after compression for the achievement of the equilibrium state.

The second step of the measure process is the heart of the CS, imposing the oscillations to the sample, by stepper - motor [12].

In the last step, the acquired data are processed to extract the necessary rheological quantities, for the characterization of the materials, including the complex modulus G^* and the loss factor $tan\delta$. Finally all required data will be saved in an *Excel* file.

IV. Tested Material

The rheological tests were performed on samples of composite polymeric material. The aim is to demonstrate that the composite material has superior properties to those of each one component, as the reinforcing phase has significantly better mechanical properties, both in terms of resistance and rigidity.

The composite materials generally are classified according to the physical structure of the reinforcement and not for its composition. In this activity were tested samples of composite material in dispersed particles, constituted by the elastomer polydimethylsiloxane (PDMS), *Sylgard 184*, with various carbon black concentrations [13], [14].

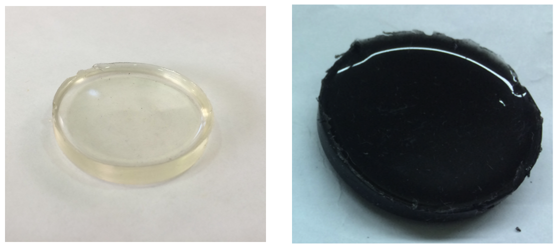

Fig. 5. Tested samples: pure PDMS and PDMS with a % of CB

Sylgard 184 belongs to the "silicones family." They are seeds of thermoplastic materials - highly crosslinked crystalline. It is characterized by a low glass transition temperature, $-125°C$, which gives it a good thermal stability compared to other polymers.

The fully crosslinked polydimethylsiloxane is a very transparent and brittle material, which crumbles at low stresses and deformations. It presents other properties, including a high permeability, good dielectric properties, weather resistance, lubrication properties, good biocompatibility, and visual clarity. *PDMS* is the most important polixanes, and used in the scientific and commercial areas. The mechanical strength of the pure polydimethylsiloxane products plays a subordinate role, for instance silicon gel for encapsulation of electronic parts, prosthesis, absorption of vibrations.

To obtain an improvement of the properties of the fracture, like tensile strength, tear strength and abrasion, and also a consequent increase in the elastic modulus, the use reinforcing fillers is necessary, usually addeding them in the non-crosslinked silicon at the production stage of compounding. The main reinforcing filler is *carbon black* [15], [16].

The reinforcement phase, dispersed within the matrix, presents basic physical and geometric characteristics to improve the mechanical and rheological properties of the final composite. To synthesize these composites was used the method of *solution blending*. This method provides initially to identify an appropriate solvent (chloroform, acetone, toluene), in which the polymer is treated in solution. The chosen solvent is used to mix with the polymer, so the suspension of the additive is dispersed in the same solvent. In the mixing phase, the surface of the additive is coated by the polymer and, after the removal of the solvent, is favored interconnection between the additive and the polymer. The carbon black was added before the beginning of the crosslinking, previously dispersed in chloroform and then added to the silicon.

The carbon black is presented as finely carbon powder, black in color, formed by particles of almost spherical shape. The carbon black particles generate the aggregates agglomerate in cluster. The particles size is a fundamental property, which does not change when carbon black is mixed in any other polymeric material. Finer particles provide a more effective reinforcement and a higher viscosity, resulting in an increase in the coagulation force, with the necessity of more energy to make possible their dispersion in a composite material.

The increase of the amount of carbon black improves the hardness and the resistance to traction of the rubber, which becomes more rigid with a remarkable wear resistance. This filler provides different physical characteristics, ultraviolet absorption and electrical conductivity, used in equipment and high-performance electronic devices. For the realization of samples, it has been designed an aluminum mold, in order to obtain the same thickness, diameter, regular and homogeneous shape. This mold allows to solidify the polymer melt by curing, even at high temperatures. In fact, the samples tested in this activity have undergone a hardening process at a temperature of $100°C$.

V. Experimental Tests

The creep and relaxation tests in the linear regime are important for the determination of the viscoelastic behavior of a material. More frequently, however, the viscoelasticity of a fluid is measured through mechanical - dynamic tests, commonly referred to as frequency response [17], [18], [19], [20], [21]. In this project the strain controlled rheometer works in SMT configuration, ie separate motor and transducer. The tests in oscillatory regime consist in subjecting the sample, placed between the two parallel plates, at a compression, depending on the percentage of the thickness specimen, and at a shear strain defining by an harmonic equation, so as to measure the resulting stress. The equation that describe the sinusoidal displacement by stepper-motor, is given by:

$$\theta_s = \theta_{s0}\, sin\,(\,\omega\, t\,) \tag{1}$$

Therefore the corresponding deformation of the sample is expressed by:

$$\gamma = \frac{\theta_s(t)}{h}\, r = \frac{\theta_{s0}}{h}\, r\, sin\,(\omega\, t) = \gamma_0\, sin\,(\omega\, t) \tag{2}$$

with r radius of sample, h its thickness, ω oscillation frequency and γ_0 maximum angular strain amplitude.

It is necessary that the sample has the same diameter of the upper plate, for a better distribution of the load, to minimize the mistakes during the measurement. The sample provides a tangential stress obtained by a transducer, measuring the torque transmitted to the upper plate by the sample:

$$M\,(t) = M\, sin\,(\,\omega\, t + \delta\,) \Rightarrow \tau = \tau_0\, sin\,(\,\omega\, t + \delta\,) \tag{3}$$

Phase angle δ represents the delay between the application of the deformation and the stress. The shear stress is represented by the sum elastic and viscous components:

$$\tau = \tau_0\, cos\,\delta\, sin\,(\,\omega\, t\,) + \tau_0\, sin\,\delta\, cos\,(\,\omega\, t\,) \tag{4}$$

dividing τ for the maximum deformation is obtained the so-called $complex\ \ modulus\ G^*$:

$$G^* = \frac{\tau_0}{\gamma_0} = G'\, sin\,(\,\omega\, t\,) + G''\, cos\,(\,\omega\, t\,) \tag{5}$$

It is possible to define G^* as the measure of the total resistance relative to the deformation of material, when it is repeatedly subjected to a shear stress. It can be estimated as the vector sum of the *storage module* G' and the *loss module* G''.

$$G' = \tfrac{\tau_0}{\gamma_0}\, cos\,\delta \qquad G'' = \tfrac{\tau_0}{\gamma_0}\, sin\,\delta \tag{6}$$

The ratio between the storage modulus and the loss modulus measures the relation between dissipated energy and the potential energy stored during a cycle, knowned as *loss factor*:

$$tan\,\delta = \frac{G''}{G'} \tag{7}$$

Referring to the rheometer used in lab, the styrene sheet with the strain gauge acts as a transducer, through which it is possible to determine the response of the tested sample in terms of torque, and therefore shear stress. Considering the scheme shown in Fig. 6, in which is shown the top view of the set constituted by the upper plate and the sheet, it is possible to derive the appropriate mathematical relationships between the physical quantities.

By equilibrium of moments in the insertion point of the sheet into the notch of the top plate, it is possibile to derive the relationship between the bending moment to which is subject the plate and transmitted torque by the sample to the upper plate:

$$\begin{cases} M_f = F \cdot b \\ M_t = F \cdot r \end{cases} \Rightarrow M_t = M_f\, \frac{r}{b} \tag{8}$$

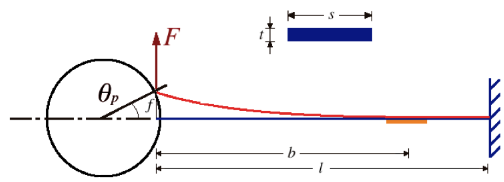

Fig. 6. Top view of the set constituted by the upper plate and the sheet

b is the distance from the center of the strain gauge at the point of application of the tangential force F, and r the radius of the sample, which coincides with the radius of the top plate.

The bending moment is represented by the equation of the calibration curve of sheet.

It is possible to determine the shear stress τ:

$$\tau = \frac{M_t}{W_t} = \frac{M_t}{I_p} r = \frac{2\, M_t}{\pi\, r^3} \tag{9}$$

VI. Analysis of Results

The rheological tests were performed on specimens composite polymeric material specimens having different concentration of carbon black. The samples have problems inherent repeatability of execution tests on the same specimen, due to the dependence of filler agglomerations by state deformation undergone [22], [23]. It was decided to adopt a standard test procedure, in the following way:

1) By stepper - motor, an angular deformation is fixed on the sample, with three different amplitudes: $5°$, $10°$ and $15°$.
2) Three different percentages of compression have been defined, in function of the sample thickness: 25%, 50% and 75%, each of which is applied for strain amplitude specified at point 1.

The protocol of each test requires that the stepper - motor takes 5 oscillations at the same frequency, for a number of times equal to 10. Furthermore, after the desired compression, a waiting time is set equal to 60 seconds, for the redistribution of the tensions into the polymer. All samples were tested at a constant ambient temperature ($T = 24°C$). The obtained experimental data are rapresented according to the angular deformation applied to the sample or at different frequencies [24].

By frequency response of the tested sample, it is possible to extract one of the rheological variables, the *complex modulus* G^*. In Fig. (7) is shown parameterized curves according to the filler concentrations and the percentage of compression applied, at a given angular deformation / frequencies, in logarithmic scal.

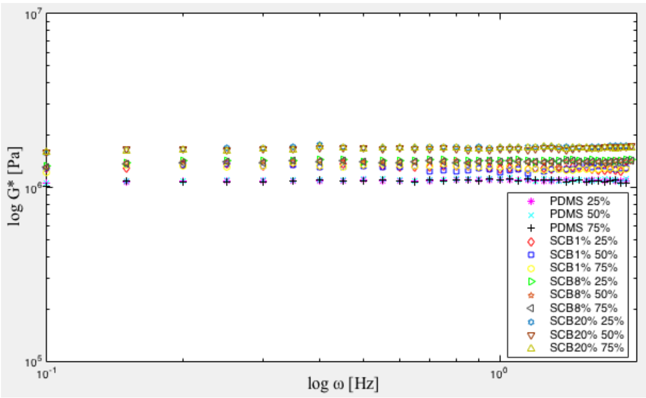

Fig. 7. Dynamic modulus G^* vs frequency

PDMS samples were tested and considered as reference for comparison with other composite materials, in order to highlight the reinforcing effect of the filler. Increasing the percentage of carbon black CB causes a consequent increase of G^*, as found in the literature.

Moreover, the dynamic modulus is independent by applied compression. By experimental data analisys, the values of complex modulus $G*$ is acquired at a specific frequency, (0.1 Hz and 1.95 Hz), varying the shear strain. In the short linear viscoelastic range (LVE) at low strain G^* is manteined constant; in correspondence of a certain shear strain value, the linear viscoelastic region end and the modulus decrease slightly. Furthermore this critical value of deformation assumes values lower than the decreasing of the concentration of carbon black. With the addition of carbon black, the composite material become more rigid and viscous, with a consequent increasing of the module G^*, with also the probability of deterioration at low deformations, causing a reduction of the LVE region.

Some scientists have observed that for elastomers enriched with carbon black, there was no presence of agglomerations induced by deformation [25], [26]. However, if this phenomenon is verified, the curve of the complex modulus G^* presents a maximum point at the low deformation region, rather than a plateau. In order to form this plateau, it is necessary that create the filler agglomerates in the microscopic structure of the sample. Since the reinforcing phase is dispersed randomly, it can form a single agglomeration due to the low filler concentrations. This phenomenon could occur in a short average distances between the aggregates, since their densification result in the formation of a crosslinked structure. The heterogeneity of the material is also due to the size of the agglomerates, which increases with the applied load.

By amplitude strain sweep is also possible to observe a frequency dependence, with an increase of the complex modulus, for each concentration of filler.

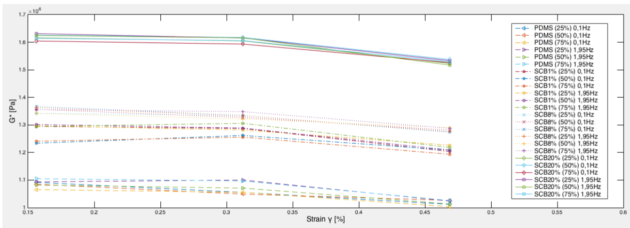

Fig. 8. Dynamic modulus G^* vs shear strain

To analyze the variation of viscoelastic response for the tested material is represented in terms of shear stress, plotted vs. time and applied to different amplitudes of angular deformation.

In Fig. (9), at fixed frequency (1Hz), the increasing amplitude of deformation is observed and also a proportional increasing of the stress undergone by the tested specimen. The curves are shifted, showing the viscoelastic behavior of the

VII. Conclusion

The aim of this paper was to verify the accuracy and reliability of a rotational rheometer with parallel plates, made

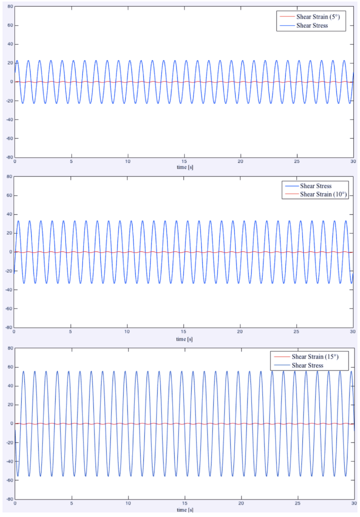

Fig. 9. Stress response to varying strain amplitudes.

in Heavy Equipment Mechanics Laboratory of the University of Catania, with the implementation of hardware and software components. To evaluate their effectiveness were tested PDMS samples enriched with different concentrations of carbon black.

The dynamic tests were performed subjecting the samples to a standard oscillatory regime, which allowed to obtain the rheological characteristics of the elastomers viscoelastic behavior. The performed study is focused on the evaluation of the complex modulus G^* to varying of the working frequency and the adopted filler percentages

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