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AUTOMATIC SYSTEM FOR MICROMECHANICAL PROPERTIES ANALYSIS

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Abstract.- This paper gives the description of an automatic and programmable device especially designed for the analysis of the mechanical properties (dynamic modulus of elasticity, creep and stress relaxation) of samples under light loads. This microprocessor controlled device is a torsion pendulum operating in forced oscillations in the very low frequency domain (V.L.F.: 10^{-11} Hz). Its characteristics are compared with those of other existing equipment and some typical results illustrate the operating range of this pendulum. This set up can be associated to a viscoelasticimeter working in tension-compression, shear, flexion, in the range 7.8 Hz - 1000 Hz.

1. Introduction.- The investigation of the dynamic modulus of elasticity makes it possible to analyze the atomic or molecular motions occurring during the strain of the material. The dynamic modulus, stress ε and strain σ ratio can be written in the form:

\[ G^* = G \epsilon^\phi \]

where \( \phi \) is the phase between stress and strain. Tan \( \phi \) can also be called the internal friction coefficient.

The measurement techniques of internal friction often make it possible to obtain the spectral \( G \) and tan \( \phi \), plotted versus temperature in a quasi constant frequency domain. In the case of a temperature dependent processus, the Arrheni's law allows to easily establish a total equivalence between the change in temperature and the change in measurement frequency. The situation is very different with glass solids because the molecular motion (and all related macroscopic properties) changes in a more complex manner with temperature, and the relaxation time distribution is generally very wide; in this case, it is essential that both methods be used: the measurements with variable temperature (fixed frequency) and the isothermal measurements (variable frequency). Besides, in the last case, it seems possible to study the material in a determined and constant structural state: the lower the temperature (and consequently the measurement frequency) the easier it is to obtain the constant structural state; and this is of peculiar interest, when the spectrum shape of the relaxation times changes with the thermal history of the material.

In fact, in order to increase the informations, it is always better to shift the spectra towards the low temperatures, and to work in a frequency domain as low as possible.

Therefore a device working in forced oscillations is required.

Such systems have been suggested for a long time in literature to study viscoelastic properties of polymers (1) (2) (3) (4) (5) (6). Recently a V.L.F. torsion pendulum has been designed to measure internal friction of metal materials (7), then applied to polymeric materials; unfortunately the mechanical characteristics of this set-up...
does not make it possible to study correctly a sample, the stiffness of which decreases by several orders of magnitude, leading to inaccurate results (8) (9). The programmable device described herein makes it possible to analyze automatically the internal friction and modulus spectra by frequency and/or temperature sweep in the range \((10^{-5} - 1 \text{ Hz}; 100 \text{ K}, 520 \text{ K})\).

It is characterized by its capacity to measure the internal friction \(\tan \phi\) by several orders of magnitude (from \(3 \times 10^{-4}\) to about \(10\)), and by its ability to measure the changes in modulus by about 4 orders of magnitude. The solicitations are various: beyond the sine solicitation (applied strain amplitude or applied stress amplitude) necessary to study the spectra by harmonic analysis, a constant stress (creep test) or a constant strain (stress relaxation test) can be applied to the sample.

The relative strain amplitude to which the sample is subjected varies from a few \(10^{-6}\) to a few \(10^{-4}\).

Furthermore, the whole assembly is presently improved in order to obtain with the same electronic unit the driving of either the torsional pendulum or a viscoelasticimeter working in tension-compression, shear or flexion in the range of 7.8 Hz to 1000 Hz.

Presentation

The set up consists of two basic parts (10)
- a mechanical unit (fig.1) which allows to apply a torsional stress to the sample. This sample is located in a cryostat regulated and programmed in temperature, depending on the investigating range.
- an electronic unit (fig.2) controls the mechanical unit and cares for date and spectra computation.

Mechanical Unit

The sample represents the resilience torque of an inverted compound torsion pendulum with a very low moment of inertia. The natural frequency of this pendulum lies usually between a few Hz and some hundred Hz; therefore the inertia effects can be neglected, when the operating frequency stays around one Hz.

When the sample modulus changes by several orders of magnitude during a test, the pendulum system suspension must be carefully considered. The apparent stiffness \(K_a\) of the oscillating part is not only that of the sample, but is equal to:

\[
K_a = K_s + \frac{K_m K_e}{K_e + K_m}
\]

\(K_s\) : torsional stiffness of the suspension ; \(K_m\) : combined torsional stiffness of frame and transmission ; \(K_e\) : torsional stiffness of the sample.

The equipment is sufficiently rigid (this has been checked by linking together both the fixed and free jaws) to assume the condition \(K_m \gg K_e\), and therefore \(K_a \approx K_s + K_e\) in all cases. With metal samples, where the modulus remains high during a test, one assumes further that \(K_s \ll K_e\) (with a possible correction).
On the contrary, with non crystalline solids, the modulus of the sample can vary a lot during a test, and the influence of the suspension can lead to incorrect results, because $K_e$ can be inferior to $K_s$, which yields $K_a \approx K_s$ (this point has been mentioned in the introduction).

Consequently the suspension has been designed with a tungsten wire of very small diameter ($\phi 30 \mu m$), making $K_s$ much smaller than $K_e$, even when the decay of the sample modulus is spread over more than 3 orders of magnitude. Therefore, the equivalence of $K_a$ to $K_e$ is insured over the entire operating range of our equipment.

- **Electronic unit**

  Built around a microprocessor, the electronic unit plays two roles:
  - it generates the coils excitation current producing the torsional stress applied to the sample, according to a law programmed by the user before each test.
  - it processes the results of the strain measurement with respect to this stress, and gives for each period the complex modulus (modulus $G$ and phase angle $\phi$).
a) **Sine current generator**

In order to generate a very low frequency signal, the digital synthesis is the best method. For this purpose the period is divided in a great number of steps \( n \) (here \( n = 4092 \)).

Besides this generator can apply, either the stress or the strain. In the first case the current (i.e. the torsion torque and consequently the stress) is controlled from the reference, while in the second case strain is taken as reference signal.

By disabling the master clock and acting on the offset control, creep or stress relaxation tests can be performed.

b) **Microprocessor system and date processing**

The C.P.U. (Z 80 8 bits microprocessor) includes 4 K Bytes of RAM and 2 K Bytes of ROM, which contains the software. A fixed or floating point 32 bits arithmetic unit (AMD 9511) reduces the program size and ensures a high computation speed. Results are stored on a floppy disk unit. The system works with conversational programs through an interactive terminal (CENTRONICS 761 KSR). The automatic frequency sweep, the computation of the dynamic modulus for each frequency, and the output of the results on printer and plotter are carried out by the program.

With the definition of the complex modulus, it is possible to write in complex notation:

\[
\sigma = G^* \varepsilon = Ge^{j \phi} 
\]

with: \( \sigma = \sigma_M \cos \omega t \); \( \varepsilon = \varepsilon_M \cos(\omega t - \phi) \) and \( G = \sigma_M / \varepsilon_M \).

Practically drifts can occur and extra noise can be superposed to the theoretical values. Besides the signals can show some DC offset. Thus if \( \sigma_0 \) and \( \varepsilon_c \) are both electrical voltages representing the stress and strain respectively, we can write, in the case of an applied stress:

\[
V_{\sigma} = K_1 \sigma = V_1 \cos \omega t ; \quad V_{\varepsilon} = K_2 \varepsilon = V_2 \cos(\omega t - \phi) + at + b + n(t) \quad \text{and} \quad G = \frac{V_1}{V_2} \left( \frac{K_2}{K_1} \right) = \frac{V_1}{V_2} x K 
\]

where \( at \) is the drift, assumed to be linear with time in a first approximation; \( b \) is the zero offset, \( n(t) \) a noise assumed gaussian. The problem at stake is to determine the characteristics \( V_2 \) and \( \phi \) of \( \varepsilon_c \), knowing the reference signal \( \varepsilon_r \). As a matter of fact, the voltages \( \sigma_0 \) and \( \varepsilon_c \) are sampled, simultaneously, at a rate of 4092 steps per period, and in step with the generator master clock; so the date processing implies the set of the sampled values \((\sigma_0^*, \varepsilon_c^*)\) in place of \((\sigma_0, \varepsilon_c)\).

3. **Calibration.** - If the loss angle \( \phi \) can be directly obtained without problem it is not the case for the modulus \( G \), because the constant \( K_a \) interferes. In order to determine this constant in the case where \( K_a = K_e \) (cf. mechanical unit). The reference sample with a known modulus has to be previously tested. In absolute, the error attached to the measurement of the modulus is estimated at about 10 \%, it is mainly due to the determination of the geometrical form factor.
On the other hand, relative changes smaller than 1% can be easily detected.

4. Application examples and conclusion. - This section deals with the results obtained with some experiments using some inherent characteristics of the above-described set-up.

a) Internal friction measurements (and modulus changes measurements) versus temperature. As it is the usual way of work of this equipment we will only give some references for this kind of measurement [11, 12].

b) Complex modulus measurements versus frequency:

Polymeric materials: two types of polymeric materials have been tested, one organic (atactic polystyrene, 120000 in molecular weight, Fig.3), the other inorganic (glassy selenium, fig. 3). The behaviors versus temperature of both materials are comparable: in the glassy transition zone (= 30°C for selenium, and ≈ 100°C for polystyrene), a very high and wide tan φ peak can be observed associated with a decay of the modulus by several orders of magnitude. It represents the classical mechanism of the principal relaxation in the polymeric materials presenting a rubbery plateau, more or less apparent. These results are not in agreement with those presented in literature (see (8) and (9)) for the above mentioned reasons in the introduction.

Oxide glass (fig.4): Above the glass transition zone, this type of glass presents a decay of the modulus and a regular increase of the internal friction versus temperature (12).

c) Creep and stress relaxation tests under light load. A glassy sele-
ium sample has been subjected to a creep test (13), then to a stress relaxation test (fig. 5). The quick response of the set-up allows the creep and relaxation curves to be used a few tenth of a second after loading.

d) Measurements performed with the viscoelasticimeter: In order to improve the set up here presented, by increasing the range of frequency of measurements a viscoelasticimeter can be connected with the same electronic unit. An example of results obtained in flexion in the case of reinforced polyester is shown in fig. 6 (13).

As a conclusion, the here-in presented equipment enables measurement of the internal friction and the elastic modulus of materials as different as metals, glasses and polymers, and this possible over wide frequency and temperature ranges, and in a large operating range, for the values of the complex modulus (G' changes by more 3 orders of magnitude and tan φ from 10^-3 to 10). Moreover, the same sample can be subjected to various tests, without additional manipulation: measurement of the elastic modulus, creep and stress relaxation.

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