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(54) APPARATUS FOR MEASURING THE DYNAMIC VISCO-ELASTIC PROPERTIES OF NON-LIQUID, VISCOUS COMPOUNDS, INCLUDING GELS

(71) We, SOCIETE NATIONALE DES CHEMINS DE FER FRANCAIS, a French Body Corporate, of 45, Rue Saint Lazare- 75436 Paris Cedex 09, France, GILLES SAUVAGE, of 9 Rue Jean XXIII- 91800 Brunoy, France, and ROBERT SAGLIER, of 2 Allee Michel-Ange, 91400 Alfortville, France, both citizens of the French Republic, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention relates to an apparatus for measuring the dynamic visco-elastic properties of viscous, non-liquid substances, including gels. The invention also concerns a testing method utilising this apparatus.

It is known that it is very difficult to effect the measurement of the visco-elastic properties of certain flexible and malleable products, in view of the inherent physical properties of these products, these products including lubricant greases, gels, natural or synthetic elastomers or rubbers.

The present invention has the object of providing an apparatus for measuring these intermediate compounds between the "genuinely" solid and liquid products.

The apparatus according to the invention is characterised in that it comprises, mounted in an insulated jacket or casing in which circulates a fluid at a given temperature, a vessel or container intended to take the product to be tested, means for imparting vibratory stresses to the product and, mounted on the vessel, a displacement-detecting device cooperating with a reference or target plate placed on the surface of the product to be tested.

The apparatus according to this invention also preferably comprises a thermal probe permanently installed in the vessel containing the product to be tested and, if necessary, a device for stirring the product to be tested, as well as a device emitting radiation.

The present invention also concerns a testing method characterised in that a sample of a product to be tested is placed in the apparatus of the invention and is subjected to a process for the measurement of its dynamic visco-elastic properties by means of vibratory stresses imparted by a conventional vibrator device cooperating with the apparatus of the invention, in which case the amplitude responses detected by the pick-up or detector cooperating with the target and reference element are compared with those obtained after the sample has been subjected to various thermal, shearing or radiation stresses, the different comparative measurements being carried out at a predetermined temperature.

The present invention will be better understood from the following description of the non-restrictive embodiments of a measuring apparatus, with reference to the accompanying drawing in which:

Figure 1 is a sectional view of the apparatus according to the present invention;

Figure 2 is a partial view of the apparatus in Figure 1 along another axial section;

Figure 3 is a partly sectional view of another embodiment of the apparatus, which can be used in particular for testing elastomeric compounds;

Figure 4 is a partial sectional view of a further embodiment according to the invention, provided with a stirring device;

Figure 5 is a response graph of a substance before testing, and

Figure 6 is a response graph of the substance corresponding to the graph in Figure 5, but after testing.

The container 1 holding the product to be tested, for example a grease, mounted

fluidtightly by way of a seal 12 in a jacket 2 surrounded by heat insulation 4. A fluid at a given temperature θ, which is supplied through an inlet pipe 5 and discharged through an outlet pipe 6, circulates between the jacket 2 and the container 1. The jacket 4 is secured to a base plate 15*a* by a set of screws 7, which plate 15*a* is itself firmly secured to a frame 13 by screws 14.

A detecting system provided with a proximity detector or sensor 3 is arranged axially relative to the container 1 and is surrounded by a detector guide-support unit 10 which fits onto the aperture of the container 1. This detector 3 is connected by plugs to a suitable device capable of handling the data detected, and cooperates with a reference or target plate 16 (in Figure 3) of the metal pad type placed on the surface of the product to be tested.

A thermal probe 16 (see Figure 2) immersed in the product to be tested is positioned in an orifice 18 which passes through the wall of the container 1. This thermal probe 16 is provided with plugs 17 intended to connect said probe to a suitable display or indicator appliance.

The spindle 19 (see Figure 4) of a propeller-like stirring device 20 passes through a cover plate 21 of the container 1 and can be so arranged that, when the spindle rotates, the propeller 20 can impart shearing stresses to the test product placed in the container 1.

In the embodiment of the apparatus according to Figure 3, the container 22 has a flat bottom 22*a* designed to receive parallelepipedal samples of the substances to be tested, for example a block of elastomer E, on whose upper face is arranged the target or reference element 16 which cooperates with the detector 3.

By utilising the apparatus according to the invention, it is possible for a sample of the test product to be subjected to the combined (or not) action of various strains or stresses, for example of temperature, shearing, vibrations and/or radiation. It is possible, for example, to obtain a temperature which can be adjusted by about one degree centigrade, within a temperature range of between −40 and 150°C, by passing a suitable fluid through the insulated jacket 2.

The rotating agitator 19, 20 may attain a speed which can be adjusted, for example, within a range of from 10 to 3000 rpm, so as to subject the substance to the desired shearing stresses. Similarly, a vibratory motion can be applied to the test product along any one or more axes, the former being adjustable both in amplitude, for example within a range of from 0 to 5mm, and in frequency, for example within a range of from 0 to 10 kHz.

A method of measuring the elastic properties of a lubricant grease will now be described below, solely by way of example.

In this test there is determined the response of the compound to vibratory stresses produced, for example, by means of a suitable commercial vibrator device, the values being read on a visual indicator or recorded, or else by a combination of these means.

The curves shown in Figure 5 are obtained using an appropriate precision measuring instrument. For the solid-line curve the test is conducted at a temperature of 90°C and at 20°C for the broken-line curve. These curves are plotted for the amplitude responses (in μm) to the stresses of vibrations having different excitation frequencies (in Hz) for an excitation amplitude of ± 6mm and frequencies of from 0 to 60 Hz.

The graph in Figure 6 shows the response curves of the preceding examples tested under the same conditions as previously but after they have been subjected for several hours to the action, combined or not, of the temperature previously set at a given level, of shearing or of radiation. In view of the detachable nature of the detector support in the apparatus according to the invention, these different tests can be performed *in situ* by replacing the detector device with a fluid-tight cover, stirring device or radiation-emitting appliance.

On the response curve shown in solid line in Figure 6 a peak is reached followed by a decline, the ascending portion ahead of the peak being "less steep", moreover, on the y-axis in relation to the corresponding curve in Figure 5. This phenomenon is even more obvious if a comparison is made between the steepness of the broken-line curves in Figures 5 and 6 respectively.

This method of measurement makes it possible to determine whether or not there exists one or more resonances for the frequency range investigated, as well as the evaluation of the 'unctuousness" of the grease tested. Unctuousness means the property exhibited by certain lubricants of adhering to a greater or lesser extent to the components which they are intended to lubricate.

A simple visual examination of the curves in Figures 5 and 6 enables significant conclusions to be drawn with regard to the influence of various parameters, for example frequency and temperature, on the behaviour of the grease to be tested.

Mathematical analysis of the physical phenomenon of forced harmonic vibration, to which the sample is subjected during the test, makes it possible to determine two parameters which are characteristic of dynamic behaviour, i.e. the modulus of elasticity and viscosity (unctuousness and internal damping).

The modulus of elasticity can be calculated with a satisfactory degree of approximation using the formula

$$\Sigma = \frac{\rho}{16h^2 F_1^2}$$

where ρ is the density of the grease, h is the height of the sample to be tested and F_1 is the value of the primary resonance frequency.

The testing method, which can be carried out by means of the apparatus according to the invention, makes it possible:

- to distinguish a new product by its elasticity (in μm) and also by its resonance frequency;
- to evaluate the combined effect of the parameters of stresses such as temperature, rotation, vibrations, as well as, optionally, the effect of certain parameters, dependent upon the manufacture of the product, on its consistency or mechanical strength;
- to measure the unctuousness of a lubricant by determining at which value of the force applied to the test specimen the latter becomes detached from the walls to which it adheres in the knowledge of this force ($F = m\gamma$) and also of the wall areas of the test vessel, it is thus possible to determine the stress or strain at which there occurs the loss of adhesion of the lubricant to the walls of the vessel.

WHAT WE CLAIM IS:-

1. An apparatus for measuring the dynamic visco-elastic properties of a viscous, non-liquid substance comprising a vessel for containing the substance to be tested, means for imparting vibratory stresses to the substance, and a displacement-detecting device mounted on the vessel, the said detecting device cooperating with a reference or target plate able to be placed on the surface of the substance to be tested, wherein the vessel is mounted in an insulated jacket through which a fluid at a given temperature can be circulated.
2. An apparatus as claimed in claim 1 wherein the container is provided with a thermal probe.
3. An apparatus as claimed in claim 1 wherein the detecting device is associated with a stirring or shearing device.
4. An apparatus as claimed in claim 1 wherein the detecting device is associated with a radiation-emitting appliance.
5. An apparatus for measuring the dynamic visco-elastic properties of a viscous, non-liquid substance substantially as hereinbefore described with reference to and as illustrated in either Figures 1 and 2 or in Figure 1 and 2 as modified by Figure 3 or Figure 4 of the accompanying drawings.
6. A method of testing utilising the apparatus as claimed in a claim 1 comprising placing a sample of a substance to be tested in an apparatus as claimed in claim 1, subjecting the sample to a process for measuring its dynamic visco-elastic properties by means of vibratory stresses imparted by a conventional vibrator device which cooperates with the said apparatus and comprising comparing the amplitude responses detected by the detector cooperating with the target and reference element with those obtained after the sample has been subjected to various thermal, shearing or radiation stresses, the different comparative measurements being carried out at a predetermined temperature.
7. A method as claimed in claim 6 substantially as hereinbefore described.

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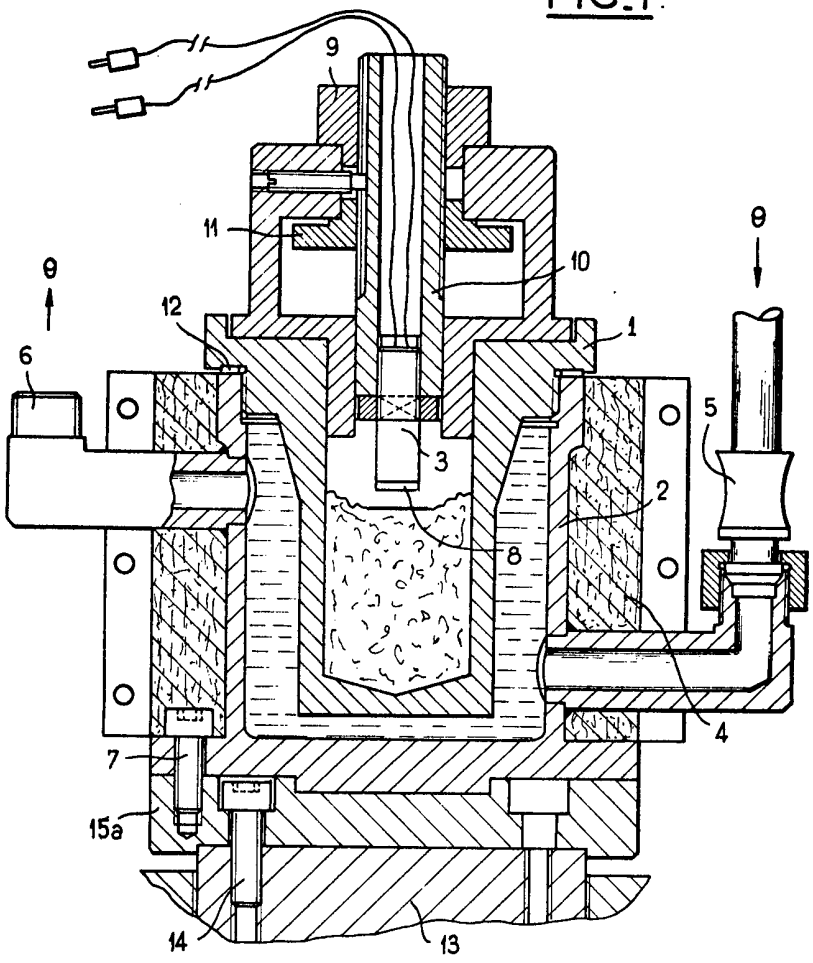
1593828 COMPLETE SPECIFICATION

3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

FIG.1.



1593828 COMPLETE SPECIFICATION
3 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheet 2

FIG_2

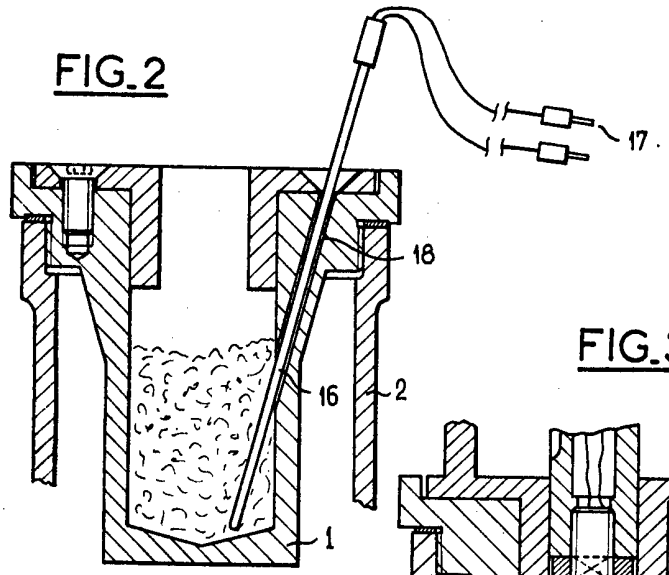
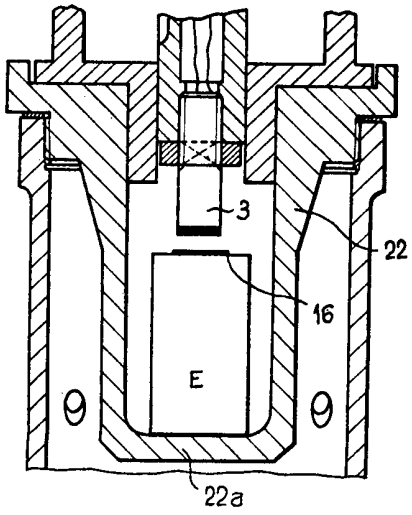


FIG_3



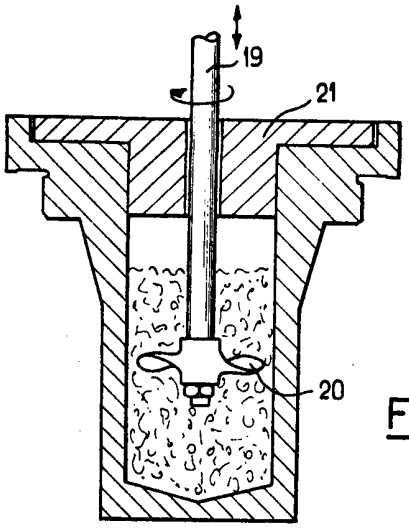


FIG_4

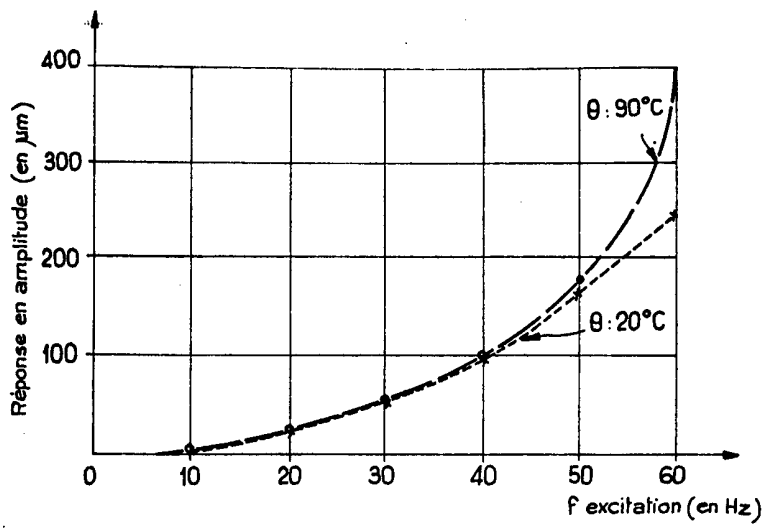

Réponse en amplitude (en µm)
400
300
200
100
0
10
20
30
40
50
60
θ : 90°C
θ : 20°C
f excitation (en Hz)

FIG_5

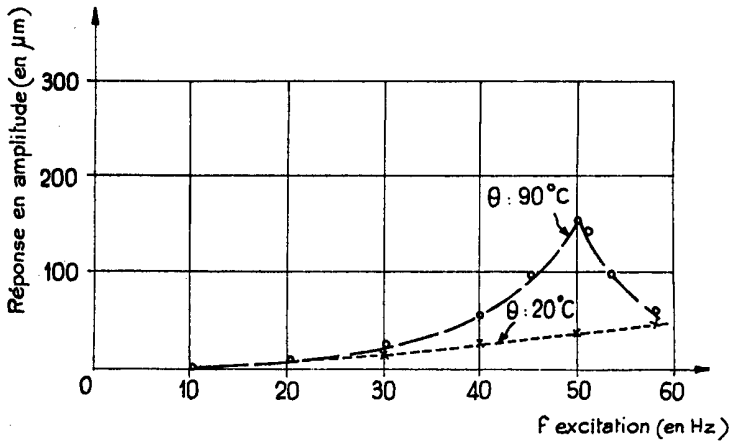

Réponse en amplitude (en µm)
300
200
100
0
10
20
30
40
50
60
θ : 90°C
θ : 20°C
f excitation (en Hz)

FIG_6