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INSTRUMENT FOR MICROTHERMOMECHANICAL ANALYSIS

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ABSTRACT

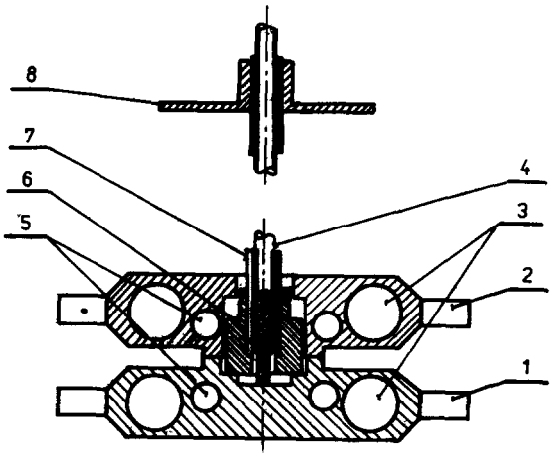
A new instrument for microthermomechanical analysis has been devoloped, possessing high accuracy for diversifier stress of test specimen. The instrument finds multilateral application in the complex assessing of polymer properties over a wide range of temperatures.

Thermomechanical analysis utilizes various instruments [1]. Results gained are closely related to the sophistication of devices that have been used. The essential problem lies in attaining exact states of temperature. This problem was dealt with in our laboratories and a measuring apparatus, which is described in this contribution, was designed to verify fields where the differential thermomechanical method could be employed [2].

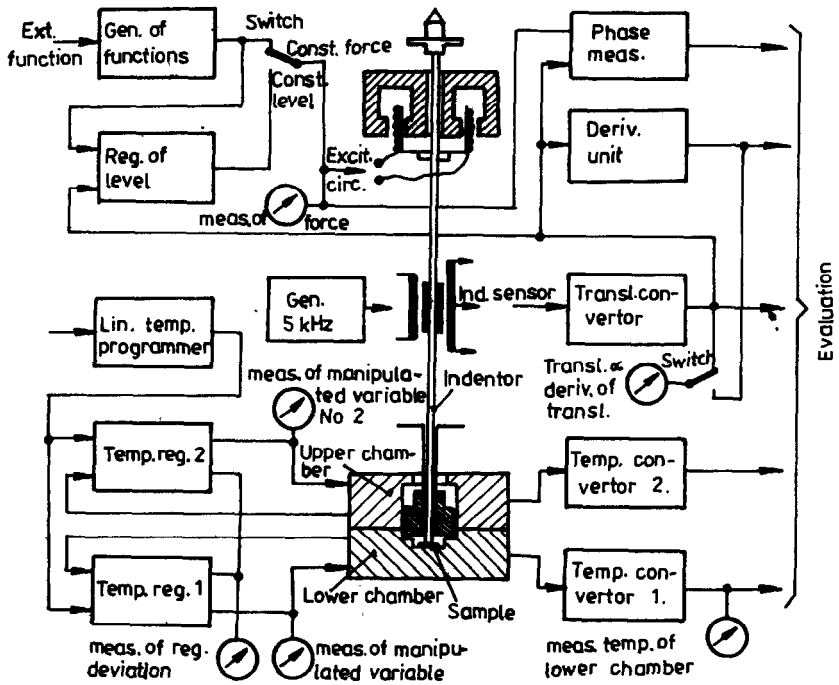
Prime attention in construction of the apparatus aimed at elimination of influences reducing measuring accuracy. These mainly involve heat transfer between the heat source and measured sample, and thermal dilation of the measuring apparatus.

In order to secure maximum agreement of temperatures between heated chamber and sample, the choice was made to employ microsamples thickness 1 mm, diameter 4 mm in direct contact with the metallic wall of heated chamber. This solution enabled us to measure at relatively good temperature agreement between heated chamber and sample, up to the range of temperature increments approaching 10 ^{o}C/min. Samples of thickness less than 0.3 mm allow even faster increases in temperature.

Construction of the heated chamber for containing sample, together with the indentor, is shown in Fig. 1. The heated chamber is divided into two parts. The measured sample is put into lower part 1 under indentor 4. The chamber is designed stepwise (lower and upper diameters 4 mm and 8 mm resp.), which makes it possible to carry out thermomechanical tests in the solid state. A beam-shaped test piece, which must be longer than 4 mm, is placed into the stepped chamber, forming thus a beam 4 mm long supported on both



Design of heated chamber for containing sample
Fig. 1



Simplified block scheme of thermo-analyzer
Fig. 2

ends. Edges of the beam can be tightly fastened by pressing ring 6. The value of pressing force can be selected by means of a weight placed on plate 8. The upper chamber 2, which is heated to the same temperature as the lower chamber, serves to prevent heat losses through indentor 4, pressing ring 6 and connecting tube 7. Holes 3, symetrically situated in both halves of the heated chamber, are designed to contain electrical heating and holes 5 to contain platinum resistance thermometers.

A simplified block scheme of the apparatus is shown in Fig.2. Heating of both chambers to the desired temperature proceeds separately through temperature regulators 1 and 2. Desired temperature is requested through a temperature programmer in the range of 0 to 400 °C. A linear temperature increase can be selected in discrete values of 1 to 9 °C/min, multiplied by factors 0.1, 1, 10. Each chamber is provided with two platinum resistance thermometers. One is connected to the mentioned temperature regulator, the other to a linearized temperature convertor. Outputs from this serve for registration and evaluation.

Translations of the indentor are measured by means of a translation induction sensor of transformer type. The sensor is powered from a stabilized generator of harmonic pulses 5 kHz. Translation of indentor is evaluated in the translation convertor which is adjusted to the range of ± 1 mm, with corresponding unified output signal ± 10 V. In order to facilitate evaluation of measured parameters, the translation signal may be derived in a deriving unit with adaptable deriving factor. The real translation of indentor, or its derivation, is indicated in a panel measuring instrument.

The generator of functions is designed to select basic mode of action of the thermomechanical analyzer, i.e. "constant force" or "constant level".

At "constant force" the indentor is under the action of constant force generated by a magnetoelectric member. Adjustment is carried out by means of d.c. generating current passing through a coil. The adjusted pressing force ranging from 0 to 1 N remains constant over the whole working translation of the indentor. A variable force of 0 to 0.5 N adjustable amplitude that is shaped rectangular, triangular or sinusoidal according to choice, with a fre-

quency of 0.005 to 5 Hz, may be superposed on the d.c. component.

At "constant level" the indentor is adjustable to arbitrary level and maintains this constantly by means of a regulator of level. A change in indentor level occurs only in case force necessary to maintain the determined level exceeds ± 1 N. The indentor level may also be modulated by rectangle, triangle or sinusoid shaped pulses of maximum range ± 0.5 mm. The frequency of these pulses may be continuously adjusted over range of 0.005 to 5 Hz.

In order to evaluate phase relations between the force acting on indentor and level of indentor, there is a convertor designed to measure phase (0 to 360°). The component of force can be increased by means of a weight up to a maximum value of 5 N.

The whole evaluation device is complemented with auxiliary circuits for calibrating and gauging records, which makes digital treatment of the results possible.

The described instrument has multilateral use. The test specimen can be stressed by pressure, bending and shear. The course of stress may be time invariable or time dependable in accordance with selected function. These broad possibilities provide for a complex assessment of thermomechanical properties in a wide range of temperatures. One potential use is the subject of an independent contribution [3].

REFERENCES

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