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Abstract

Full Text

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PHYSICAL CHEMISTRY

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A METHOD FOR DETERMINING POLYMER TRANSITION TEMPERATURES FROM THE DEFORMATION OF POWDERS

(Presented by Academician V. A. Kargin, July 30, 1963)

One of the most convenient ways of evaluating the mechanical properties of polymers at various temperatures is the method of recording thermomechanical curves, first proposed by Kargin and Sogolova (^{1}). The essence of the method consists in determining the deformability of a polymer body under the action of an external mechanical stress transmitted to the specimen through a punch, the area of whose contact with the specimen is considerably smaller than the area of the specimen.

Usually polymers are studied by this method in the form of monolithic specimens prepared from a polymer block. However, it is often necessary to carry out thermomechanical tests of polymers obtained in the powder state.

The literature contains statements that thermomechanical curves by the Kargin and Sogolova method can also be recorded for polymers in the form of powders. The erroneousness of this opinion has been considered in detail in (^{2},^{3}). In this connection, a powdered polymer intended for thermomechanical tests should be converted into a monolith. In most cases the monolith is prepared by pressing the polymer powder at a temperature above T_g. The monolithic character of the specimen is, as a rule, not evaluated. In working with amorphous polymers, the transparency of the specimen is often taken as an indication of its monolithic character. However, in the general case, the transparency of a specimen does not characterize the degree of its monolithic character (^{4}). It should also be borne in mind that the process of monolithization of most polymers is accompanied by irreversible changes leading to a change in the transition temperatures.

Fig. 1. Schematic diagram of the apparatus:
1 –die, 2 –punches, 3 –temperature-measurement point

A distinctive feature of the method we have developed for determining the transition temperatures of polymers is that the thermomechanical curve is constructed

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Fig. 1. Schematic diagram of the apparatus: 1 —die, 2 —punches, 3 —temperature-measurement point

Figure 1: Fig. 1. Schematic diagram of the apparatus: 1 —die, 2 —punches, 3 —temperature-measurement point

Figure 2

Figure 2: Figure 2

from the deformation of the polymer powder, without converting it into a monolith. The polymer is tested in a cylindrical press mold with an internal diameter of 10 mm, shown schematically in Fig. 1. A 100 mg portion of polymer powder is placed in the cold press mold between the punches, one of which has a hole 1.5 mm in diameter. After this the powder is compacted under a pressure of 320 kgf/cm^2; the load is then reduced to 40 kgf/cm^2, and the polymer is heated at a rate of 1° per min. At specified temperature intervals the deformation is determined from an indicator with a scale division of 0.01 mm.

Figures 2–4 show deformation curves for powders of a number of polymers. In the initial temperature range, thermal expansion of the powder occurs mainly, and only slight compaction takes place, owing to the fact that the compacting pressure during preliminary tableting is several times higher than the working pressure. In the glass-transition region, considerable compaction of the powder is observed, caused by the transition of the polymer into the highly elastic state. The glass-transition temperature is taken to be the temperature corresponding to the intersection of the tangent at the point of inflection with the smallest ordinate. The magnitude of the deformation in the highly elastic state is determined by the ratio between the increase in the height of the specimen due to thermal expansion and its decrease due to compaction of the grains of the polymer powder. When the polymer passes into the viscous-flow state, it begins to penetrate into the opening present in the punch, as a result of which a sharp rise is observed on the curve. As shown in Fig. 2, the temperature of transition into the viscous-flow state is taken to be the temperature at the point of intersection of the linear portions corresponding to the highly elastic and viscous-flow states.

Fig. 2. Deformation curves of powders of polyvinyl chloride (1) and polymethyl methacrylate (2)

It should be noted that the transition temperatures determined from the thermomechanical curves of monolithic specimens and from the deformation curves of powders coincide. An important feature of the powder-deformation method is that this method makes it possible to determine the glass-transition temperatures of crystalline polymers (Figs. 3 and 4), whereas in the thermomechanical method, at the loads usually used, the high elasticity of such polymers, as a rule, does not appear. This high sensitivity of the method is due to the differ-

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Figure 3

Figure 3: Figure 3

Fig. 4. Deformation curve of polycarbonate powder

Figure 4: Fig. 4. Deformation curve of polycarbonate powder

ence in the mechanical properties of bodies in the monolithic and powder states. When polymer powders are heated, the integral value of the deformation within individual grains and the displacement of these grains relative to one another is measured. Consequently, the powder system proves to be a kind of "amplifier" of deformation, which, apparently, is the reason for the high sensitivity of the method.

Fig. 3. Deformation curves of powders of amorphous (1) and isotactic (2) polystyrenes

The study of the transition temperatures of polymers from the deformation of powders is especially important for polymer-processing practice, since it makes it possible–

makes it possible to trace the behavior of the powder-like system over a wide temperature interval.

Thus, by the method of studying the deformation of powders, it is possible to determine the transition temperatures of polymers without converting them into a monolith. This is especially important when it is impossible to obtain a monolithic specimen from a polymer powder, and also when preparation of a monolith is accompanied by irreversible changes in the polymer. The high sensitivity of the method makes it possible to determine, by this method, the glass-transition temperatures of crystalline polymers.

Fig. 4. Deformation curve of polycarbonate powder

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