STUDY OF THE PRESSURE DISTRIBUTION OVER ROTATABLE BRIDGMAN ANVILS

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UDC 539.31:620.17

Study has been made of the pressure distribution over rotatable Bridgman anvils prepared from KhVG steel and VK-6 alloy, working at room temperature and over the pressure range from 10 to 40 kbar. The pressure distribution was determined by following the polymerization of acrylamide under the combined action of high pressure and deformational shear. The polymer yield was found to increase with increasing pressure at fixed angle of rotation of the anvil. Study was made of the effect of pressure on the polymer yield over various annular zones marked out within the sample. The pressure distribution was found to vary with the material of which the anvil was constructed, the normal pressure, and the sample geometry. It was also found that there was one interval within which the pressure over the anvil was almost constant.

Devices using the Bridgman anvil are widely used in high-pressure studies, especially those involving high-pressure deformational shear.

These devices are unsatisfactory insofar as they do not assure uniformity of pressure distribution over the working sample. A knowledge of this distribution and its dependence on such factors as the nature of the anvil material, the treatment to which this material was subjected, and the sample geometry, is essential, not only for improving the technique of working with these devices, but also for correcting results reported from various physical and chemical studies on phase transitions and reaction kinetics and mechanisms.

Data on the pressure distribution over fixed anvils have been given in [1-4]. Experiments with diamond anvils [2] showed the pressure to be higher at the center than on the periphery of the sample, while exactly the opposite results were obtained in experiments with tungsten carbide anvils [5]. Experiments with tempered steel anvils [6] showed center pressures 24-70% below mean pressure in thin-sheet samples, and 20-55% above in thick-sheet samples.

There is essentially no available information concerning the pressure distribution over rotatable anvils. It has been suggested that [4] the distribution here would not be the same as for fixed anvils, but possibly more nearly uniform. Measurements of the shear stress on anvils of various diameters [7] have been interpreted as indicating center pressures lower than the pressure on the edges of the sample.

The present study aimed at acquiring information on the pressure distribution within the sample, and the variation of this distribution with the pressure and the nature of the anvil material. Experiments were carried out on acrylamide samples. The extent of polymerization of the acrylamide proved to be independent of the time of anvil rotation, being determined, at fixed temperature, by the pressure and the displacement coordinate of the sample section [8]. The polymerization rate constant was measured over zones located at various distances from the sample center, and the pressure in each zone then calculated from the known pressure dependence of the rate constant.

Experiments were carried out with an anvil made from KhVG steel (hardness R_C = 62-64), 20 mm in diameter, and an anvil made from VK-6 alloy (hardness, R_A = 88). The purified acrylamide was pressed

Moscow. Translated from Zhurnal Prikladnoi Mekhaniki i Tekhnicheskoi Fiziki, No. 1, pp. 143-147, January-February, 1974. Original article submitted June 5, 1973.

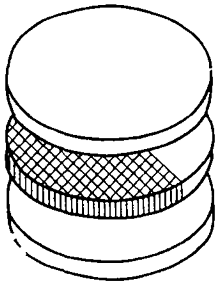

Fig. 1

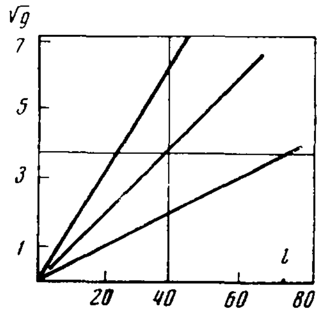

Fig. 2

into tablets, 20 mm in diameter and of the desired thickness, using a die on a 5 ton press. The tablet thickness was determined before and after each experiment using a micrometric indicator accurate to within $\pm 1\ \mu$. Each sample was subjected to the combined action of high pressure and deformational shear and then divided into three zones of 0-10, 10-15, and 15-20 mm diameter with the aid of a template. The polymer yield in each zone was determined by the method of [8]. Before the data could be used for determining the pressure distribution, it was necessary to prove that movement proceeded uniformly through the entire sample, that is to say, to eliminate the possibility of either preferred displacement along certain planes or anvil slippage under the sample. Tablets built up of two parts, one colored by the addition of 0.05-0.1 wt.% diphenylpicrylhydrazyl acrylamide, were used in studying this point.

On the KhVG anvil (surface cleanness, ∇8), and at pressures less than 5 kbar, these tablets showed no displacement of the colored interface under anvil rotation, the anvil slipping under the entire sample surface. Lagging of the peripheral zone with respect to the center set in at 6 kbar on the KhVG steel anvil, and at 10 kbar on the VK-6 alloy anvil. Peripheral zone slippage was indication of a nonuniformity in the pressure distribution, the pressure being lower at the edge than in the center of the sample under these conditions. At pressures in excess of 10 kbar on the KhVG steel anvil, or in excess of 20 kbar on the VK-6 alloy anvil, the angular displacement of the colored interface proved to be identical with the angular rotation of the anvil. By working with three-layer tablets of the type shown in Fig. 1, and carrying out microscopic section studies, it was proven that movement took place over the entire tablet and not just over one, or even several, slip planes.

When working on the KhVG steel anvil at a pressure of 10 kbar, and at percentages of acrylamide polymerization q of the order of 40-50%, $\sqrt{q}$ proved to be related to l, the angle of anvil rotation, through a linear equation of the form (Fig. 2)

$$\sqrt{q} = K'(l + l_0) \tag{1}$$

l_0 being an effective angle equivalent to sample flow resulting from compression on the anvil, and K' a constant which varies on passing from zone to zone, but is independent of the angle of rotation.

We will now derive an equation relating the relative polymer yield and the zone radius. Let it be supposed that the yield q and the angle l in a tubular section with wall thickness dr are related by Eq. (1). The physically significant factor here is not the angle of anvil rotation, but rather the section displacement |x| which, for deformational shear, is given by the equation

$$x = lr / h \tag{2}$$

r being the radius, h the length of the elementary tubular section in the sample, and x the relative displacement of the sections with respect to one another.

Using (2), an equation analogous to (1) can be obtained, namely

$$q = K\,[(x + x_0) / h]^2 \tag{3}$$

K being a constant independent of both the angle of anvil rotation and the zone radius.

The amount g of polymer formed over a elementary tubular section of radius r, length h, and wall thickness dr is then given by the expression:

$$g = 2\pi rhq dr = 2\pi rhK\,[(x + x_0) / h]^2 dr = 2\pi r^3 K\,[(l + l_0)^2/h] dr \tag{4}$$

TABLE 1

Pressure, kbar	Anvil material	Mean sample depth in zone, $\cdot 10^{-2}$, μ			Constant $K \cdot 10^6$			Calculated pressure, kbar		
		zone diameter, mm			zone diameter, mm			zone diameter, mm		
		0–10	10–15	15–20	0–10	10–15	15–20	0–10	10–15	15–20
10	KhVG steel	1	0.9	0.7	1.82	1.67	1.44	12.7	10.9	7.8
20	KhVG steel	1	0.9	0.7	3.24	4.16	5.00	14.7	19.8	23.8
20	VK-6 alloy	1	0.9	0.7	3.80	3.66	2.30	24.8	24.0	14.3
30	VK-6 alloy	1	0.9	0.7	4.96	4.28	3.88	33.0	30.0	27.9
40	VK-6 alloy	1	0.9	0.7	6.00	5.80	6.92	38.8	38.1	41.8

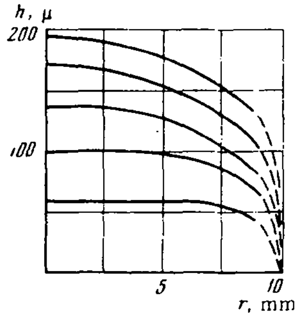

Fig. 3

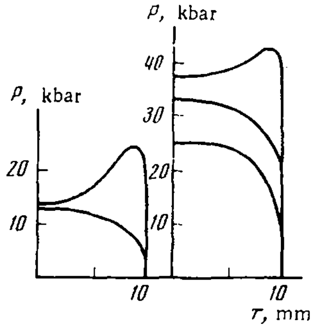

Fig. 4

The total weight G of polymer formed in a tubular section of wall thickness $(r_2 - r_1)$ is finally obtained by integrating Eq. (4) with $(l + l_0)$ = const and h = const, the result being

$$G = \int_{r_1}^{r_2} 2\pi \left[(l + l)^2/h\right] K r^3 dr = \frac{\pi}{2} K \left[(l + l_0)^2/h\right] (r_2^4 - r_1^4) \tag{5}$$

The relative polymer yield is by definition $q = G/G_0$, G_0 being the total amount of polymer formed in the tubular section as given by

$$G_0 = \pi (r_2^2 - r_1^2) h, \quad q = G / \pi (r_2^2 - r_1^2) h = K (l + l_0)^2 \cdot (r_2^2 + r_1^2) / 2h \tag{6}$$

The constant K of Eq. (6) was evaluated from the data of Fig. 2 (cf. Table 1). Since the coordinate of section displacement is a function of the sample thickness, a correction for the variation of this factor on passing from zone to zone was introduced in Eq. (6). The zone thickness was obtained from the mean of 50 sample geometry measurements performed at the end of experiments at various pressures and with various anvil rotations (Fig. 3). These measurements showed the tablet form to be weakly dependent on the pressure, over the interval from 10 to 40 kbar, and on the angle of rotation, over the interval from 5 to 60°.

The data of the table make it clear that the constants for the 15-20 mm zone increased more rapidly with increasing total pressure than did the constants for the central zone; and, of course, the same must have been true of the pressures in these zones themselves. Such data can, naturally, be expressed directly in terms of pressures once the rate constant vs pressure relation is known. The latter can, in turn, be developed from the relation between the integral polymerization rate for the entire sample and the pressure, the latter given by the equation

$$\lg (K_P / K_{10}) = aP - b \tag{7}$$

in which P is the pressure, in kbar, $a = 2 \cdot 10^{-2}$ kbar^{-1}, b = 0.2, and K_{10} is the polymerization constant for a 10 kbar pressure.

Equation (7) and the relative values of the constant were used to estimate pressure in the various zones of the sample. The results of these calculations are shown in Fig. 4 as pressure distribution probability profiles. Here the pressure maximum is seen to shift from the center to the periphery of the sample, with increasing total pressure and with passage from the steel to the alloy anvil. The observation of a pressure maximum at the center of the sample has been repeatedly reported from work with fixed anvils, and is consistent with the results of theoretical calculations in which perfect anvil rigidity is assumed [9, 10]. The anvil deforms only slightly at low pressures and the pressure distribution is then determined solely by sample flow under pressure. The fact that the pressure maximum shifts from the center to the periphery with rising pressure could indicate more extensive deformation at the center than on the periphery where there is support from the mass of material in the nonworking zone of the anvil. This could be considered an instance of the Bridgman mass support principle. The passage of the pressure maximum toward the center on substituting the VK-6 alloy anvil for the KhVG steel anvil could possibly be explained in the same way, the high Young modulus of this alloy assuring that anvil deformation would have minimal effect on the pressure distribution at a working pressure of 20 kbar.

It could be anticipated that the pressure distribution would be altered by changing the tablet thickness, the effect of sample properties on the distribution increasing, and that of the anvil deformation diminishing. For this reason the position of the pressure maximum is displaced toward the center of the anvil by such increases. The validity of this conclusion was confirmed by the results of experiments with tablets of various thickness, determining the polymer yield in the central and peripheral zones and, at the same time, measuring the sample dimensions. Values of the ratio of polymer yields in the central and peripheral zones are shown below. Clearly this ratio increased with increasing thickness of sample, the increase becoming even more pronounced when correction was made for differences in zone depths. With original tablet thickness of 60, 100, 150, 200, and 250 μ, the ratios of corrected polymer yields in central and peri-peripheral zones ($l = 20°$, P = 10 kbar) were respectively 0.32, 0.36, 0.44, 0.80, and 1.10.

Summarizing, it can be said that the pressure profile at fixed sample dimensions varies with the pressure, pressure maximum being located at the sample center at the lower pressures and moving toward the periphery as the pressure is increased. At fixed pressure, the position of pressure maximum is displaced toward the center as the sample thickness is increased.

Passage from KhVG steel to VK-6 alloy anvils carries to higher pressures that pressure range over which the position of the maximum is displaced from the center to the periphery of the sample. For a given anvil pair and given sample type, there proved to be one pressure interval over which the pressure distribution showed minimum deviation from uniformity. This opens the possibility of so selecting the anvil material and sample geometry as to assure closest approach to pressure uniformity in Bridgman devices. The pressure distribution on fixed structural steel anvils is similar to that observed on KhVG steel anvils at 20 kbar. This suggests that the pressure distribution is not affected by movement of the anvil.

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