The influence of vibration on sedimentation stability of polymer composite materials

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Abstract. The work is devoted to the study of the effect of vibration on the creation of a gradient layer in cast polymer composite materials. The influence of vibration frequency and vibration exposure time on the distribution of filler particles in a polymer matrix has been studied. The presented results can be used in the manufacture of cast parts for the needs of mechanical engineering from polymer composite materials with an increased content of particles of the dispersed phase.

1. Introduction

Among the various forms of physical and mechanical action on colloidal systems, which include liquid metal and polymer composite materials, vibration occupies a special place as one of the effective means of creating a controlled dynamic state of dispersed systems [1-3].

The main distinguishing feature of vibration is the ability to transfer energy to the system of high specific power at a small amplitude of its displacement during the period of oscillation. At the same time, the possibility of regulating vibration parameters (frequencies and amplitudes) over a wide range makes it possible to extend its effect to large volumes of the processed dispersed system.

Vibration can be considered as a universal form of mechanical action on dispersed systems in general and on structured systems, which include composite materials, in particular [4]. That is why vibration has found widespread application in various industries, including technological processes for the production of cast products for mechanical engineering [5-7].

Since vibration significantly increases the fluidity of solid-liquid suspensions, it has been widely used in recent years in the manufacture of moulds from chemically hardening sand-resin mixtures. It is known that vibration significantly affects not only the fluidity, but also the surface tension at the interface between the liquid and solid phases [8], which significantly improves the wettability of the surface of the casting mould with a liquid polymer composition.

Since liquid polymer compositions with an increased content of the dispersed phase have not only reduced fluidity, but also low filling capacity [9-12], it was decided to apply vibration in the technology of manufacturing cast models from polymer composite materials in order to improve the quality of the surface imprint. As a result, a significant improvement in the quality of the imprint of the model surface was achieved. Meanwhile, the imposition of a vibration effect on the polymer composition could not but affect the sedimentation stability of the latter, namely, the distribution of particles over the volume of the polymer base. On some samples, significant stratification of the polymer suspension was noted after vibration treatment.
In this regard, the purpose of this work was to study the effect of vibration on the sedimentation stability of polymer compositions.

2. Methods

Sedimentation stability, or in other words, the ability of particles of a dispersed phase not to settle in a dispersion medium, is described by the Perrin formula

\[
\lg \frac{n_0}{n_1} = 0.58 \pi g (\rho - \rho_0) r_{av}^3 (h_1 - h_0) \frac{N_A}{RT}
\]

where \(n_0\) and \(n_1\) are the number of particles at the sample height \(h_0\) and \(h_1\); \(\rho\) and \(\rho_0\) are the density of particles of the dispersed phase and dispersion medium (liquid polymer - the matrix of the composition); \(r_{av}\) - average radius of particles; \(g\) - acceleration of gravity; \(N_A\) is Avogadro's number; \(R\) is the universal gas constant.

For colloidal particles, the settling process is balanced by the diffusion process. As a result, sedimentation equilibrium arises. In polymer composite materials with a particle size much larger than colloidal ones (on average, from 0.01 to 1.0 mm), sedimentation equilibrium cannot be achieved in principle. However, due to the significant viscosity of the polymer base of the composition, a so-called pseudo-equilibrium is observed in the newly prepared volume of the suspension for some time, the duration of which significantly depends, among other things, on the viscosity of the compound.

The sedimentation stability of colloidal solutions, which include liquid polymer compositions, is traditionally estimated with some approximations using the Stokes equation

\[
v = \frac{2}{9\eta} r_{av}^2 (\rho_1 - \rho_0) g
\]

where \(v\) is the sedimentation rate of particles of the dispersion phase in a dispersed medium (filler particles in a liquid polymer); \(\eta\) is the viscosity of the liquid polymer.

The above dependence is successfully applied not only for approximate practical calculations, but also for the analysis of the sedimentation process. In accordance with equations (1) and (2), the sedimentation rate of particles increases with an increase in the difference between the densities of the filler and the compound \(\Delta \rho = \rho_1 - \rho_0\), the particle size \(r_{av}\), and decreases with an increase in the viscosity of the compound \(\eta\). Moreover, the most significant effect on sedimentation stability is exerted by the particle size, since it is present in the formula in the second degree, as well as the base viscosity. Deviations from the calculated parameters are mainly explained by the deviation of the particle configuration from the spherical one, since the indicated formula was obtained precisely with this approximation taken into account.

It is known that vibration can, on the one hand, significantly reduce the viscosity of a liquid composition, and on the other, increase convective movements in it. A decrease in viscosity contributes to the stratification of the composition, and convective movements, on the contrary, to mixing and averaging of the composition. Thus, it is not possible to theoretically unambiguously evaluate the effect of vibration on the sedimentation stability of a liquid polymer composition.

Samples for research were prepared on the basis of a cast polyurethane compound using powders of metallic and non-metallic materials. To obtain a polyurethane compound, the calculated amount of polymer component A was measured, the catalyst and the corresponding amount of component B were introduced into it, and thoroughly mixed using an overhead stirrer "Pro Spin" DW-90 with a variable shaft speed of up to 3000 rpm. Mixing of fillers (powders) was carried out as follows: after mixing component A and the catalyst, the necessary additives were introduced in a calculated amount, thoroughly mixed for 1-5 minutes, then component B of the polymer was added and mixed for 0.5-3 minutes until a homogeneous mass was obtained. The resulting mass was poured into polypropylene tubes with an inner diameter of 17 mm and a length of 150 mm with an end sealed on one side.
In this work, we used:

- liquid polyurethane compound ADV 13-2 according to TU 2226-046-227369360-99, density 1.26 g/cm³;
- aluminium powder PA-4 GOST (Russian standard) 60-58-73;
- iron powder grade PZhV2 GOST 9849-86;
- crystalline graphite of GL grade in accordance with GOST 5279-74.

To study sedimentation stability, a device was designed that provides constant frequency and amplitude of oscillations during the solidification of a polyurethane compound with a filler. The installation for studying the sedimentation stability of polymer composite samples consists of four columns, on which a massive load-bearing plate, 500x300x15 mm in size, weighing about 16 kg is suspended by cables. A DC motor with a power of 150 W and a variable speed of 2950 rpm is attached to the plate from below. A distinctive feature of the engine is the shaft exit to both sides of the housing. On these protruding parts of the shaft, eccentrics weighing 70 g were fixed to create imbalance and vibration during engine operation. The samples were mounted on the upper side of the plate using special clamps. This ensures, firstly, a rigid contact of the samples with the plate (this is necessary for transmitting vibration energy impulses to the sample), and, secondly, to prevent displacement or inclination of the samples during the experiment.

The vibration frequency was controlled photometrically. The vibration frequency of the vibrating unit in the experiments was constant and amounted to approximately 160-170 Hz, the vibration amplitude was about ± 1.0 mm.

The vibration treatment time was varied by changing the time of the onset of solidification of the images. Accordingly, the time of the onset of solidification (time of loss of vitality) was quite accurately (± 5 min) controlled at intervals of 30 min in the range from 30 to 180 min by changing the amount of catalyst. After this time, the liquid compound solidified within 2-3 minutes and the sedimentation process was terminated. Complete solidification of the samples occurred a little later: on average, 4-6 hours after the start of the experiment.

After complete solidification of the compound, the obtained samples were removed from the installation and examined. Thin sections were made from the obtained samples of polymer composite material (PCM). To do this, they were removed from the vibration unit, taken out of polypropylene tubes and sawn in height into five equal parts: at a distance of 30, 60, 90, 120, and 150 mm from the lower end surface. The sawing was carried out at a speed of 15 mm/s on a Polisib P80A electric saw perpendicular to the direction of sedimentation. The resulting sample fragments were processed on a Griender Polisher P22 grinding machine with a table rotation speed of 300-400 rpm to obtain the required surface quality of the sections. Sandpaper of various grain sizes was used for grinding. For the best removal of scratches and gouges from the saw, sanding was started on paper with a grit of P400. Sanding was completed with sandpaper with a grit of P1200 - 1500.

Sedimentation resistance was studied metallographically by examining the structure of the samples. The structure of the obtained samples was studied using a Nikon EPIPHOT 200 optical microscope at x25 magnification. On each microsection, the number of inclusions per 1 mm² was calculated.

3. Results and discussion

The most pronounced results were obtained with a holding time of 60 min. The resulting dependences of the distribution of filler particles over the height of the sample are presented in Figures 1 - 3.
As follows from the data presented, the sedimentation process is observed in all samples, without exception. Figure 1 shows graphs of changes in the concentration of various fractions of particles of iron powder PZhV2 along the height of the samples. At the base of all samples, without exception, the presence of a completely metallized layer was found, which indicates a significant intensity of the process and is explained by the large difference between the densities of the filler material and the base ($\rho_{ADV} = 1.26$ g/cm$^3$, $\rho_{PZhV2} = 7.8$ g/cm$^3$). However, the process of sedimentation of particles of different iron fractions proceeded in different ways. As follows from the data presented, in samples with a larger size of fractions of 0.4 - 0.63 and 0.63 - 0.8 mm, an almost complete separation of the filler and base material is noted. In samples with particles of fractions of average size, an almost linear dependence of the change in the concentration of particles along the height was noted. In the upper part of the samples with a particle size of less than 0.1 mm, a deviation from the linear dependence towards an increase in sedimentation stability was noted.

Figure 2 shows the graphs of changes in the concentration of particles of various fractions of PA-4 aluminium powder along the height of the samples. According to the data obtained, the sedimentation process in all the samples proceeded almost similarly to the samples with iron powder, but with a lower intensity, which is explained by the significantly lower difference in the densities of the base and filler ($\rho_{ADV} = 1.26$ g/cm$^3$, $\rho_{PA4} = 2.7$ g/cm$^3$). Nevertheless, in samples with a size of fractions of 0.63 - 0.8 and 0.8 - 1.6, complete separation of the filler and base material was also noted. Samples with powder particles of 0.16 - 0.2 mm fraction showed significant sedimentation stability: in percentage terms, the difference in particle concentration between the top and bottom of the samples is practically insignificant.
The results of studies of the effect of vibration on the sedimentation process of graphite particles of different crystalline fractional composition are shown in Figure 3. Analysis of the results obtained during the experiments showed the following: samples of polymer compositions with graphite powders of different fractions showed more significant sedimentation stability. Sedimentation of graphite is not pronounced. In particular, there is no clear separation between the base and the filler material. The graphs are almost linear. The number of particles near the surface layer of the lower end of the sample differs from the upper one by only 80 - 100 units, this is explained by the fact that crystalline graphite sediments poorly, because the filler material and the base have close density values ($\rho_{ADV} = 1.26 \text{ g/cm}^3$, $\rho_{GL} = 2.1 \text{ g/cm}^3$).
4. Conclusions
As a result of the research, it was established that in liquid polymer composite materials (PCM) based on the polyurethane compound ADV-13 with a filler in the form of iron powder PZhV2 of various fractions (from 0.1 to 0.8 mm), the superposition of vibration leads to accelerated stratification of the composition due to a significant difference in the densities of the filler and the base, regardless of the particle size.

The effect of vibration on the sedimentation stability of polymer composite materials based on the polyurethane compound ADV-13 with filler in the form of PA-4 aluminium powder and crystalline graphite of different fractions is ambiguous. Liquid PCMs with particles of both aluminium and graphite, more than 0.2 mm in size, are prone to delamination, and PCMs with particles less than 0.2 mm show fairly satisfactory stability and can be used for the production of cast products with imposed vibration.

Thus, in the manufacture of cast parts for the needs of machine building from polymer composite materials with an increased content of particles of the dispersed phase and the imposition of vibration, it is possible to recommend a PCM with a filler in the form of PA-4 aluminium powder and crystalline graphite GL with a particle size of less than 0.2 mm.

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