COMPLEX VISCOSITY OF A KAOLIN CLAY

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Abstract – The complex viscosity of a material is a two-component quantity, comprising real and imaginary parts. The real part of the complex viscosity is oftcn very useful because in many materials it approaches the ordinary steady-flow viscosity at low frequencies. Because many materials with high viscosities are very slow in reaching a steady-flow condition, the determination of the steadyflow viscosity may be very difficult; however, an approximation can often be obtained from lowfrequency values of the real part of the complex viscosity. In this study, the complex viscosity of a Georgia kaolin has been determined by measurements made on specimens subjected to oscillatory simple shear. over three decades of frequency. Other independent variables in the study are the water content of the clay and the shear strain amplitude. Data were obtained from experimental measurements in the form of values of the magnitude, or absolute value, of the complex viscosity, and the phase angle between the imposed oscillatory strain and the stress response. Empirical functional relationships are developed to relate these quantities to the independent variables, and these are in turn used to obtain the real and imaginary parts of the complex viscosity as functions of the independent variables. The results of this study indicate that the complex viscosity is not linear, but decreases approximately as a power function of the strain amplitude; the relation between the complex viscosity and the water content is approximately an inverse logarithmic one, and changes very rapidly at water contents near the liquid limit; and the phase angle increases with increasing strain amplitude approximately as a power function.

INTRODUCTION

THERE are two important types of anomalies which may appear in the mechanical properties of a material. Stress anomalies occur when strain or strain rate depends on stress in a manner other than a direct proportionality. and time anomalies occur when stress depends on both strain and strain rate together, as well as higher time derivatives of strain. In general, both kinds of anomalies will coexist to some degree in all materials. Evaluation of these anomalies is important in determining the macroscopic mechanical properties of a material for direct applications; in addition, some information on the nature of the microscopic material behavior can often be inferred from the macroscopic behavioral characteristics.

In the work reported herein, the complex viscosity of a Georgia kaolin clay is determined from measurements made on specimens subjected to oscillatory simple shear. The independent variables in this study are shear strain, frequency of oscillation, and water content, whereas the dependent variables are shear stress and phase angle. Despite observed stress anomalies, the response is expressed in the terminology of linear viscoelastic theory; in terms of response functions, the real and imaginary components of the complex

viscosity, as well as its magnitude, are examined to determine their dependence on frequency of oscillation, shear strain amplitude, and water content.

TEST PROGRAM

M aterial investigated

The tests were performed on samples prepared from a white Georgia kaolin clay, designated as "Hydrite R," which was supplied by the Georgia Kaolin Company. This clay has a grain size distribution such that· 97 per cent of the particles are smaller than 5μ and 60 per cent are smaller than 1μ . Some of the index properties are: liquid limit 53 per cent, plastic limit 35 per cent, shrinkage limit 32 per cent, and specific gravity 2·61. The X-ray diffraction and differential temperature graphs indicate that kaolinite is the only mineral present in significant quantity.

Mixtures of the kaolin clay and distilled water were prepared at nominal water contents of 45, 50, 60, 70, 80, 90, and 100 per cent. The mixtures were blended as thoroughly as possible by hand, then sealed in jars and stored in a high-humidity room for several months prior to testing; at the time of testing, the samples were found in most cases to have water contents 2-5 percentage points below the nominal value. The final degree of saturation is not known and cannot be presumed to be 100 per cent.

T est apparatus

The primary apparatus used in this test program was the Weissenberg rheogoniometer with a 6°, 5 cm cone-and-plate sample holder. This equipment, together with a custom-made modification to improve strain measurement, has been described in detail by Franklin and Krizek (1968). One important advantage of the cone-and-plate sample holder lies in the relative homogeneity of imposed stresses and strains.

Mode of testing

All specimens were tested under a sinusoidally varying shear strain of amplitude γ_0 and radian frequency of oscillation ω , imposed by rotation of the lower platen, and the resulting steady-state periodic shear stress of amplitude τ_0 was measured at the top platen; in addition, the phase angle δ or time lag Δt , as determined from the zero axis crossings of the respective curves, was measured. In order to partially evaluate the effect of loading history on the response of this clay, two modes of testing were employed: in the first mode, the oscillation input amplitude was fixed, while the frequency of oscillation was varied in discrete steps; in the second mode, the frequency of oscillation was fixed, while the strain amplitude was varied. The former tests are termed "constant amplitude" tests, while the latter are referred to as "constant frequency" tests. As will be seen subsequently, the difference in steady-state response due to these two loading histories is negligible within the ranges tested; good duplicability was obtained for all test results reported herein.

Scope of test program

In addition to the water content range of nominal values from 45 to 100 per cent, test variables were shear strain amplitude, varying from approximately 0·0006 to 0'015, and frequency of oscillation, varying from 0·0095 to 9·5 cpst with most tests performed at 0·95 cps. The initial state of the specimen is considered to be its state after placement in the sample holder, and any residual stresses are ignored. The clay tested was flocculated and is considered to exhibit negligible shear thinning or shear thickening characteristics. A total of 39 tests were performed in an airconditioned room, where actual test temperatures ranged from 22 \cdot 2 to 24 \cdot 1°C with a mean of 23 \cdot 4°C.

THEORETICAL CONSIDERATIONS

When a material is tested under sinusoidally oscillating strains, as explained above, the steadystate response can be conveniently expressed in terms of a variety of viscoelastic response functions, such as complex modulus G*, complex compliance J^* , complex viscosity η^* , etc.; a brief review of these relations has been presented by Krizek and Franklin (1967), and more detailed treatments may be found in the works of Bland (1960), Ferry (1961) and others. To provide briefly the background for the work reported herein, the complex viscosity η^* may be defined as

$$
\eta^* = \eta' - i\eta'' \tag{1}
$$

where *i* equals $\sqrt{-1}$ and η' and η'' are, respectively, the real and imaginary components of the complex viscosity and may be written

$$
\eta' = \frac{\tau_0 \sin \delta}{\omega \gamma_0} = |\eta^*| \sin \delta \tag{2}
$$

and

$$
\eta'' = \frac{\tau_0 \cos \delta}{\omega \gamma_o} = |\eta^*| \cos \delta \tag{3}
$$

in which $|\eta^*|$ equals $\tau_o/\omega y_0$ and is termed the magnitude of the complex viscosity. The real component η' is associated with components of stress and strain rate which are in phase; the imaginary component η'' , which is imaginary only in a mathematical sense, is associated with out-ofphase components of stress and strain rate. Sometimes, the real component η' is called the dynamic viscosity; \ddagger this component is often very useful, since for many materials, such as uncross-linked polymers, tested at low frequencies, η' approaches η , the ordinary steady-flow viscosity. With increasing frequency, η' usually decreases monotonically and may reach a value many times smaller than η . On the other hand, for other materials, such as cross-linked polymers, η is infinite, but η' will approach a finite limit at low frequencies (Ferry, 1961). Since many materials with high viscosities are very slow in reaching a steady-flow condition, the determination of η may be very difficult; however, an approximation can often be obtained from the low-frequency value for η' . It should be noted that the expression of experimental data in terms of viscoelastic response

t Although frequencies are specified here in terms of cycles per second, note that frequency ω in Eqs. 2 and 3 must be expressed in radians per unit time.

 \ddagger Alternatively, $|\eta^*|$ is sometimes termed the dynamic viscosity.

functions does not necessarily imply strict validity of the associated aspects of the theory, such as transformation integrals, response spectra formulations, etc., and, in fact, these relationships should be used only with great caution.

EXPERIMENTAL RESULTS AND ANALYSIS

In raw form the experimental measurements are recorded as periodic stress and strain waveforms similar to those shown by Krizek and Franklin (1967). Data are then measured from these waveforms and formulated in terms of descriptive empirical relations. Since the effects of three independent variables are being investigated, it is necessary to adopt some systematic approach to isolate the effects of each variable, insofar as possible.

The first step will be to consider only data for a frequency of 0·95 cps and to plot in Fig. 1 the magnitude of the complex viscosity $|\eta^*|$ vs. water content *w* for various approximately constant values of shear strain amplitude γ_0 . The values at zero shear strain were obtained by extrapolating to zero strain a plot of the reciprocal of the magnitude of the complex viscosity vs. shear strain amplitude. Since the greatest rate of change with water content occurred in the vicinity of the liquid limit (53 per cent), the data representing each constant strain amplitude were fitted by parallel straight line segments with a discontinuity in slope at the liquid limit, as shown in Fig. 1.

Fig. 1. Magnitude of complex viscosity vs. water content for different shear strain amplitudes.

These lines have the equations

$$
\log_{10} \frac{|\eta^*|}{|\eta^*|_{LL}} = A(w - w_{LL})
$$
 (4)

where $|\eta^*|_{LL}$ is the magnitude of the complex viscosity projected to the liquid limit and w_{LL} is the liquid limit. The coefficient A in Eq. (4) is equal to -2.05 when the water content is above the liquid limit and -7.52 when it is below the liquid limit, and the water content and liquid limit are expressed as decimals rather than percentages. The value of $|\eta^*|_{LL}$ will be termed the projected complex viscosity and can be obtained from a complex viscosity at any water content by projecting the point to the liquid limit along a line parallel to those in Fig. 1.

With the effects of water content accounted for, the dependence of the complex viscosity on shear strain amplitude can be examined as follows. From each value of $|\eta^*|$, a value of $|\eta^*|_{LL}$ is determined according to Eq. (4), and these values are plotted in Fig. 2a against the shear strain amplitude. The curve describing the data in Fig. 2a has an equation of the form

$$
|\eta^*|_{LL} = \mathbf{B}(\gamma_0 + \mathbf{C})^{\mathsf{b}} \tag{5}
$$

where, for this particular case in which the frequency is 0.95 cps, the constants B, C, and b are

Fig. 2. Magnitude of complex viscosity and phase angle vs. shear strain amplitude

163, 0.00075 , and -1.345 , respectively. As seen in Fig. 2a, Eq. (5) describes the data very well for shear strain amplitudes up to 0.01 ; above this strain amplitude, Eq. (5) gives values of $|\eta^*|_{LL}$ which are somewhat low.

Since, in general, each of the coefficients A, B, C, and b may be frequency-dependent, the final independent variable to be studied is frequency. Although most of the tests were performed at 0·95 cps, plots similar to Fig. 1 were made at each of the seven frequencies from 0.0095 to 9.5 cps included in the test program; no systematic variation of the slopes with frequency could be detected, so the A coefficients determined for a frequency of 0·95 cps are considered to be invariant with frequency over the range tested. Additional study indicated that the coefficients C and b could reasonably be considered independent of frequency over the thousandfold range of frequencies covered. The coefficient B was found to be inversely proportional to frequency, and is given by the empirical relation

$$
B = \frac{929}{\omega}.
$$
 (6)

Combining Eqs. 4-6, we obtain the following empirical equation describing the magnitude of the complex viscosity $|\eta^*|$ as a function of water content *w*, shear strain amplitude γ_0 , and frequency of oscillation ω :

$$
log10|η*| = 2.986 + A(w - wLL) –- 1.345 log10 (γ0 + 0.00075) – log10ω. (7)
$$

Phase angle

The effects of the independent variables on the phase angle δ were studied in a systematic manner similar to the preceding. However, it was found that the phase angle is nearly independent of frequency and water content above the liquid limit, although it was found to increase very rapidly with water content up to the liquid limit. If the phase angle data from samples below the liquid limit are ignored, the effect of shear strain amplitude can be investigated by plotting phase angle versus shear strain amplitude, as shown in Fig. 2b. Under the assumption that the phase angle is zero at vanishingly small strain amplitudes (this assumption may or may not be correct, but that question cannot be resolved by the experimental evidence thus far obtained), the data can be reasonably described by the indicated curve whose equation is

$$
\delta = 360 \gamma_0^{0.39} \tag{8}
$$

where δ is in degrees. It should be noted that the value of 360 for the coefficient is the result of an empirical description of the functional relationship, and it is mere coincidence that it is also the number of degrees in a circle.

INTERPRETATION AND DISCUSSION

Knowledge of the functional relationships describing the magnitude of the complex viscosity and the phase angle allows,the complex viscosity to be determined according to Eqs. 1-3. Graphs of computed values of the real and imaginary components η' and η'' are given in Fig. 3 as functions of shear strain amplitude for various values of water content and for a constant frequency of oscillation (0·95 cps). Similar graphs would be obtained at other frequencies. The nonlinearity, or strain dependence, of the viscosity, as well as its dependence on material consistency, or water content, is immediately obvious, and resembles the behavior of many materials which show significant concentration-dependent deviations from Newtonian behavior at moderate stresses. For example, certain rubbers loaded with carbon black manifest a strain dependence for strains much lower that 0.01 (Ferry, 1961; p. 348). To provide an appreciation of the degree to which the experimental data can be described by the developed empirical equations, correlation graphs between calculated and experimental values for η' and η'' are given in Fig. 4.

The data reported herein serve to illustrate that the rheologic behavior of clays is both time- and strain-dependent. Qualitatively, the prominence of time-dependent phenomena in clays may be associated with the versatility of movement of microscopic solid clay particles coated with thin films of glue-like bound water, and the nonlinearity may be explained in terms of the particle reorientations which occur. Although, strictly speaking, a macroscopic or phenomenological measurement affords no conclusion concerning the microscopic or particulate origin of the behavior, certain speculative interpretations may be advanced.

When a material such as clay, with its particulate nature consisting of flat plate-like or elongated needle-like particles, is subjected to shear strains, certain reorientations are produced; these involve rearrangements of the positions of the particles, and the initial rheologic properties are altered. That is, the material is actually changing its structure as the strain increases, and this is manifested in the observed nonlinear response. For example, if the clay particles rotate during flow and are subjected to periodic distortions which resemble to some extent those imposed by periodic macroscopic small strain deformations, the failure of

Fig. 3. Calculated values for real and imaginary parts of complex viscosity as

functions of shear strain amplitude.

some modes of rearrangements to keep pace with the periodic changes in forces at high rates of deformation causes relatively low energy dissipation, and it is observed that there is less energy dissipated per cycle at high frequencies than low frequencies in a dynamic experiment. Accordingly, the apparent viscosity decreases with increasing rate of shear.

Beazley (1964) has reported that continued application of shear causes mechanical breakdown of the particles with a subsequent reduction in the viscosity and dilatancy of the suspension, and that the efficiency of breakdown increases with increasing rate of shear. In flocculated suspensions the rheology is determined by the forces binding the particles together in floes, by the forces holding the flocs together in a network, and by the mutual interference which may occur between floes when they move as individual entities. Such

suspensions are shear thinning, and their behavior with time at a constant rate of shear is governed by the concentration of the suspension and by the rate of shear used for the measurement. On the other hand, Beazley (1965) states that a concentrated deflocculated clay suspension might be shear thinning at low rates of shear strain but dilatant at high rates.

For many materials the steady-flow apparent viscosity exhibits a shear strain rate dependence which closely resembles that of the real part of the complex viscosity on radian frequency; in some cases, the agreement with the magnitude of the complex viscosity is better. Equation (7) does not reflect this limiting behavior, but it must be remembered that this equation is an empirical one which is applicable only over a limited range of frequencies. If the experimental measurements were obtained at sufficiently low frequencies, this

Fig. 4. Correlation between calculated and measured values of real and imaginary components of complex viscosity.

correspondence might manifest itself. On the other hand, if it were desired to obtain the instantaneous compliances of a low viscosity, very soft clay, the time scale may be such that the determination of viscoelastic behavior by a constant rate test is obscured by inertial effects, and constant frequency tests may prove essential.

Various researchers have reported work on the viscosity of clays to study the effects of degree of crystallinity, degree of flocculation, temperature, particle size and shape, shear strain rate, water content, factors influencing dilatant behavior, and other variables. Many of these studies were performed on kaolin clay specimens under constant strain rate conditions with a cone-and-plate geometry. In order to place the present work in perspective, some of the general conclusions reported by these investigators should be considered. Ormsby and Marcus (1967) found several types of rheological behavior, including Newtonian flow, dilatancy, pseudo-plasticity, and thixotropy; also, they found that for non-dilatant systems, increases in the viscosity of different particle size fractions of a single clay correlated with increased surface area, cation exchange capacity, and crystallinity, while dilatancy occurred in some fractions of intermediate particle size and was especially pronounced in certain fractions of high crystalline clays. They conclude that (i) particle size is of primary importance in controlling viscous and plastic properties, (ii) crystallinity does not generally correlate with flow properties, and (iii) the extent of dilatancy may be qualitatively correlated with the degree of crystallinity when the dilatancy is pronounced. Beazley (1967) reported that the curve of viscosity versus deflocculant dose passes through several secondary maxima and minima; however, the curves were not stable, but tended to flatten out with time.

It is also of some interest to compare the results

reported herein with the behavior of a number of polymers, as shown by the frequency response of the real part of the complex viscosity. Figure 5 shows a plot of the real part n' of the complex viscosity vs. frequency ω ; the curve for the kaolinite examined in this study is shown with data points. Curves A, B, and C represent cross-linked polymers, and curves D, E, F uncross-linked polymers, as plotted from response curves given by Ferry (1961). Some of these curves show a variation with frequency very much like that of the kaolinite over a similar range of frequencies. The polymers represented in Fig. 5 are described by Ferry as:

- (A) A *dilute cross-linked gel,* a 10 per cent gel of polyvinyl chloride in dimethyl thianthrene.
- (8) A *lightly cross-linked amorphous polymer,* lightly vulcanized Heavea rubber. cured with sulfur and an accelerator.
- (C) A *highly crystalline polymer,* a linear polyethylene with a density of 0.965 g/ml at room temperature.
- (D) An *amorphous polymer of high molecular weight,* a fractionated polyvinyl acetate of weight-average molecular weight 300,000.
- (E) An *amorphous polymer of high molecular weight with long side groups,* a fractionated poly-n-octyl methacrylate of weight-average molecular weight 3,620,000.

(F) An *amorphous polymer of high molecular weight below its glass transition temperature,* a polymethyl methacrylate.

SUMMARY AND CONCLUSIONS

In order to study the complex viscosity of a kaolin clay, a series of 39 steady-state oscillatory shear tests were conducted at water contents between approximately 45 and 100 per cent, at frequencies from 0·0095 to 9·5 cps, and at shear strain amplitudes up to about 0·015. Two types of test, "constant amplitude" and "constant frequency," were performed by means of a Weissenberg rheogoniometer with a cone-and-plate sample holder, and all data are expressed in the terminology of linear viscoelastic theory. Within the test limitations and scope of the experimental program, the following conclusions can be advanced:

- 1. The complex viscosity is not linear, but decreases with increasing shear strain amplitude approximately as the -1.3 power.
- 2. An apparent rapid change in viscosity is observed at water contents in the vicinity of the liquid limit.
- 3. The effects of two particular loading histories, constant amplitude with varying frequency

Fig. 5. Comparison of real part of complex viscosity for kaolin clay and several polymers.

and constant frequency with varying amplitude, seem to be insignificant.

- 4. The phase angle is relatively independent of frequency and water content for water contents above the liquid limit.
- 5. The real and imaginary components of the complex viscosity can be readily calculated from empirical descriptions of the magnitude of the complex viscosity and the phase angle.
- 6. The phase angle increases with increasing shear strain amplitude as approximately the 0·39 power.

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Resume- La viscosite complexe d'une substance est une quantite a deux composants, comportant des parties réelles et imaginaires. La partie réelle de la viscosité complexe est souvent utile parce que dans le cas de beaucoup de substances elle approche de la viscosité normale à écoulement stable aux basses fréquences. Parce que de nombreuses substances ayant une viscosité élevée sont très lentes à atteindre un état d'écoulement stable, la détermination de la viscosité à écoulement stable est parfois difficile. Pourtant il est souvent possible d'obtenir une approximation à partir des valeurs à basse fréquence de la partie réelle de la viscosité complexe. Dans la présente étude, la viscosité complexe d'un kaolin de Georgia a été déterminée par des mesures de specimens soumis au cisaillement oscillatoire simple, pour trois decades de frequence . D'autres parametres variables et independants dans l'etude sont la teneur en eau de I'argile et I'amplitude de I'effort de cisaillement. On a obtenu des données à partir de mesures expérimentales sous forme de valeurs de la grandeur, ou valeur absolue, de la viscosite complexe, ainsi que I'angle de phase entre I'effort oscillatoire impose et la reponse a I'effort. Des relations empiriques et fonctionnelles sont developpees dans le but de l'association de ces quantités aux variables indépendants, et ceux-ci à leur tour sont utilisés en vue d'obtenir les parties reelles et imaginaries de la viscosite complexe en tant que fonctions des variables indépendants. Les résultats de cette étude indiquent que la viscosité complexe n'est pas linéaire mais diminue de maniere approximative en tant que fonction de puissance de l'amplitude de I'effort; le rapport entre la viscosite complexe et la teneur en eau est de maniere approximative un rapport inverse au rapport logarithmique, et change très rapidement pour des teneurs en eau près de la limite liquide. Enfin I'angle de phase augmente avec une amplitude croissante de I'effort, approximativement selon une fonction de puissance.

Kurzreferat- Die Komplexviskositiit eines Materials setzt sich aus zwei Bestandteilen, einem wirklichen und einem imaginären Teil, zusammen. Der wirkliche Teil der Komplexviskosität ist oft sehr brauchbar, da er bei niedrigen Frequenzen annähernd der gewöhnlichen Viskosität im stationären Strömungszustand entspricht. Da viele hochviskose Stoffe eine lange Zeit in Anspruch nehmen ehe sie den stationären Strömungszustand erreichen, kann die Bestimmung der Viskosität in diesem Zustand recht schwierig sein, jedoch kann ein Richtwert hiiufig aus Niederfrequenzwerten des wirklichen Teils der Komplexviskosität erhalten werden. In der vorliegenden Arbeit wurde die Komplexviskositiit eines Georgia Kaolins durch Messungen an Proben, die einer oszillierenden, einfachen Schubspannung unterworfen waren, iiber drei Frequenzdekaden hinweg bestimmt. Weitere unabhiingige Variable in der Untersuchung sind der Wassergehalt des Tones und die Amplitude der Schubspannung. Aus experimentellen Messungen werden Daten für die Grösse, d.h. für den Absolutwert, der Komplexviskosität und für den Phasenwinkel zwischen aufgegebener Schwingungsspannung und Formänderungsreaktion erhalten. Es werden empirische funktionelle Beziehungen entwickelt um diese Grössen in ein Verhältnis zu den unabhängigen Variablen zu bringen, und um dann die wirklichen und imaginären Bestandteile der Komplexviskosität als Funktionen der unabhängigen Variablen zu erhalten. Die Ergebnisse deuten darauf hin, dass die Komplexviskosität nicht linear ist, sondern annähernd als Potenzfunktion der Spannungsamplitude abnimmt; die Beziehung zwischen der Komplexviskositiit und dem Wassergehalt ist beilaufig eine umgekehrte logarithmische, und ändert sich rapid bei Wassergehalten nahe der Flüssiggrenze. Der Phasenwinkel schliesslich, nimmt mit zunehmender Spannungsamplitude ungefahr als Potenzfunktion zu.

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Резюме—Комплексная вязкость материала это двухэлементная величина, содержащая истинные и мнимые части. Истинная часть комплексной вязкости часто очень пригодна, так как во многих материалах достигает она обычной вязкости стационарного поткоа при низких частотах. Ввиду того, что многие, обладающие высокой вязкостью материалы очень медленно достигают состояния стационарного потока, определение вязкости стационарного потока бывает очень трудным. Однако, можно часто получить аппроксимацию из низкочастотных значений истинной части комплексной вязкости. В настоящем исследовании, комплексная вязкость каолина штата Джорджия определяется измерениями на образцах, подвергаемых в течение трех декад частоты колебательному сгвигу. Другие независимые переменные в этом исследовании это водосодержание глины и амплитуда деформации сдвига. Данные получены были по экспериментальным измерениям в виде значений величины или как абсолютное значение комплексной вязкости иизмерениям угла сдвига фаз между наложенной колебательной деформацией и реакцией напряжения. Развивались эмпирические, функциональные соотношения, чтобы установить отношение этих величин к независимым переменным, а эти, в свою очередь, применяются для того, чтобы получить истинные и мнимые части комплексной вязкости, как функции независимых переменных. Результаты настоящего исслелования означают, что комплексная вязкость не является линейной, но уменьшается приблизительно как функция мощности ампдитуды деформации. Зависимость между комплексной вязкостью и водосодержанием приблизительно обратно логарифмическая и меняется очень быстро, когда водосодержание доходит до предела жидкости, а фазовый угол увеличивается при повышении амплитуды деформации, приблизительно как функция мошности.