DETERMINATION OF THE WORKABILITY OF CLAYS USING THE UPSET TEST

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ABSTRACT

In ceramic industry, workability (or plasticity) is generally defined as the product of yield stress and maximum deformation without fracture. In the present study, yield point values for two clay bodies have been obtained from compression tests done without friction at the die contact surface. Maximum deformations to fracture, on the other hand, have been determined from local strain measurements of bulget surfaces of cylinders compressed with friction at the die contact surface. Workability indexes have been obtained from the products of yield stresses and maximum deformations.

For both clay bodies studied, yield values decreased and amount of maximum deformation increased with increasing water content. Thus, the workability index showed a maximum determining the optimum water content.

Forming limit line (FLL) technique, based on local strain measurements on the bulget surfaces of cylinders upset with friction at the die contact surface, form the basis for evaluating the workability of metals as a function of material variables. It also forms the basis for the analysis of workability in bulk deformation processes of metallic materials. In the present case, the FLL technique has been applied to ceramic bodies in determining their maximum deformation to fracture and a new easy-to-apply technique not necessitating the use of special equipment, has been developed for the determination of workability of ceramic bodies.

INTRODUCTION

The workability (or the plasticity) is one of the most important characteristics of clays. The shaping of clays in plastic form, can be considered as one of the first material production techniques that the man has tried.

The deformation process (plastic shaping) still keeps its importance in today's technology. Ceramic products are generally put in shape by

deformation processes and the plastic shaping depends upon the plastical properties (or the additives that promote plasticity) of raw materials.

In the ceramic industry the workability is generally defined as the product of the yield stress and the amount of maximum deformation that can be obtained without fracture. The second part of this definition (the maximum fracture under the process conditions) is the only criterion that is considered in defining the workability of metallic materials. Regarding the workability of clays however, the yield stress concept is added to the definition of the workability. The reason for this is that the yield stress of clays is low. Most of the time, the shaped clay product cannot carry its own weight. Hence, a clay that has a high yield stress is evaluated to have high workability. Not being able to carry its own weight is never a problem in metallic materials so it doesn't have to be considered in the definition of workability.

Workability is not a material property by itself it also depends upon the variables of the process. For an exact workability analysis, material and process variables should be considered together. Nevertheless, the developments over the recent years have shown that these two functions can be separated from each other (1). In mass deformation processes, the forming limit lines (FLL) based on the limit strains at the instant of fracture represent the material variables, and the lines that the stress follows at the fracture areas during the process represent the process variables. In that case, the mass workability of the materials can be determined by using the FLL s regardless of the process variables. The FLL technique developed by Kuhn, Lee and Ertürk gives the amount of maximum deformation that can be obtained from the material at various strain positions under the process conditions and the process is designed on the basis of FLL s.

For the determination of the workability of clays the yield limit measurements can be performed by tension, compression and torsion experiments. For the measurement of the amount of maximum deformation however, although many methods have been developed, these are not able to represent the procedural conditions, hence they can not be used in the

procedural design. In addition, the developed procedures require special instrumentation. For example the procedure developed by Norton (3,4) requires a special instrument that applies sliding strain to rectangular samples. The aim of this study has been to develop a new procedure for the determination of the workability of clays. The procedure is based on a simple compression experiment and it consists of the application of the FLL technique which constitutes the procedural design and which can determine the workability of powdered metal and dense metal products, to clay bodies.

2. EXPERIMENTAL PROCEDURE

In order to be able to compare the experimental results and to determine the sensivity of the procedure the study has been conducted on two separate natural clays, that have different plastical properties. The Küre clay of the Söğüt region and the Deresakarı clay are materials that are known by their high and low plasticities respectively.

The clays have been broken in a conical crashing machine and a 10 kg portion of the sample has been taken from the divider as a sample. A 5 kg portion of the samples has been loaded in ball mills and ground with water for 15 hours. The aqueous suspension that has formed has been put in a plaster mould and has been kept there until it became a plastic clay. The clay has been put in and kept in plastic bags to be used later during the experiments.

Chemical analysis, rational analysis, X-ray diffraction analysis, differential thermal analysis and thermogravimetric analysis have been applied to the samples and their mineralogical formations have been determined. The physical properties of the samples have been determined by performing the wet sieve analysis and by determining the modulus of rupture, Atterberg plasticity limit, the liquid limit and the pfefferkorn plasticity water.

By the help of a steel stamp two parallel lines with a distance of 1.0 mm from each other were drawn parallel to the base of the cylinder at 3 different places on the side surfaces of the cylindirical clay samples

which were taken from a constant moisture oven and had a certain moisture level. The distance between the lines has been measured by a microscope that has micrometer readings. Cylindirical samples were placed between two iron discs which were parallel to each other. The discs and the sample were placed between the dies of the hand press. The press arm was moved as constantly as possible and the sample was deformed until fractures of 45° at the free surface of the sample could be seen by the bare eye. As soon as the fractures were observed the experiment was stopped and the distance between the parallel lines at the side surface was measured by a microscope. The diameter at the instant of fracture was measured by a compass. The limit strains in terms of axial compression $(-\varepsilon_z)$ and circuniferential tension (ε_0)

$$\varepsilon_z = \ell n \ L_f \ / \ L_0 \tag{1}$$

$$\varepsilon_0 = \ell n \ D_f \ / \ D_0 \tag{2}$$

have been calculated from expressions (1) and (2), where L_0 and L_f represent the distance between the lines at the side surfaces before and after the experiment, recpectively, and D_0 and D_f represent the diameter before and at the instant of fracture, recpectively. The samples were weighed while they were moisturized and after they have been dried at 105° during 5 hours, and the moisture percent with respect to the dry basis was determined. For every moisture interval a graph $(\varepsilon_z, \varepsilon_0)$ has been traced. Linear regression procedure was applied to the determined points and the $\varepsilon*$ values (planar strain toughness) where the forming limit line cuts the ε_0 axis were determined $\varepsilon*$ value obtained for each moisture value was plotted against the moisture percent and the maximum deformation curve has been obtained. The fractures of 45° that appeared on the samples during the compression experiment are shown in Fig 7 (a, b).

For the determination of the yield limit, the cylindirical samples with a length of 2.25 cm and a diameter (L/D =1.5) of 1.5 cm were submitted to compression between two cylindirical plates between the dies of the Instron universal instrument at a die rate of 0.5 mm/min. The mould and the touching surface were greased with vaseline and the deformations at

the side surfaces approximately up to 10 % could be prevented. The yield limits have been determined by applying the 0.2 % offset principle. Here again, the % moisture contents have been calculated with recpect to the dry amount. The workability indices for the Küre and Deresakarı clays have been obtained from the product of the yield stress and the amount of maximum deformation.

3. RESULTS

The strain curves for the Küre and Deresakarı clays obtained from the compression experiments realized under frictionless conditions, are given in Figure 1. In both of the clays the transition from elastic
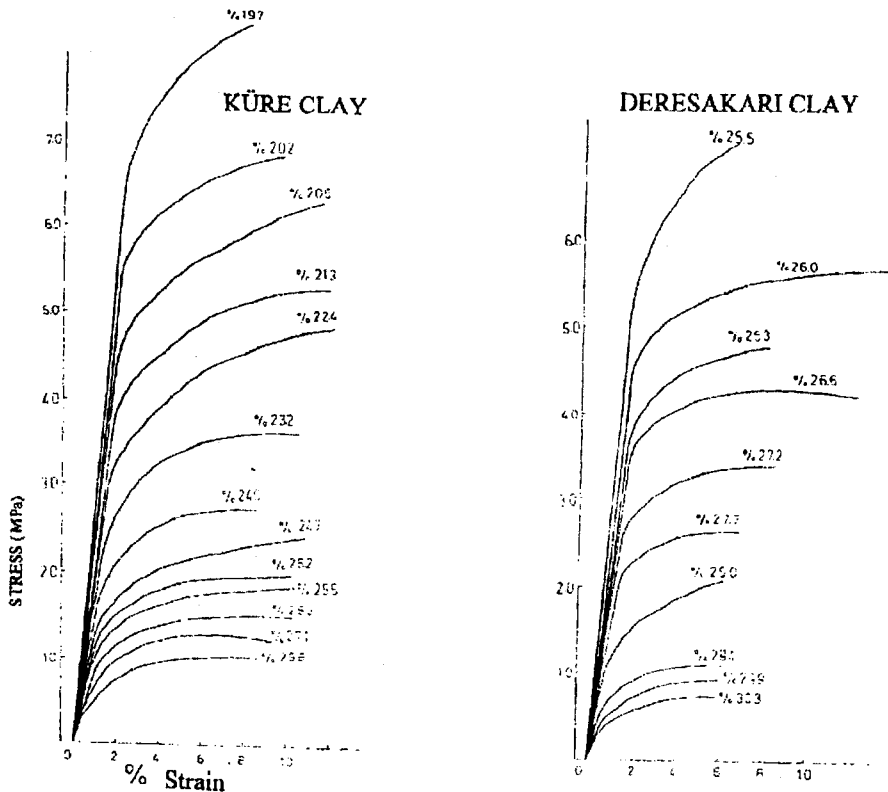

Figure 1. The stress and strain curves obtained from the compression experiments under friction free conditions for the Küre (a) and Deresakarı (b) clays.

deformation to plastic deformation is rather sharp and both clays show deformation hardening, Figure 1. The deformation hardening in the Deresakarı clay is higher than that in the Küre clay.

The workability limit lines obtained from the compression experiments under frictional conditions are given in Figure 2 (a-k) for the Küre clay and in (l-r) for the Deresakarı clay. Under the experimental moisture contents the slope of the FLL is -1/2 and the intercepts on the y-axis (planar strain toughness) increase as the moisture content increases.
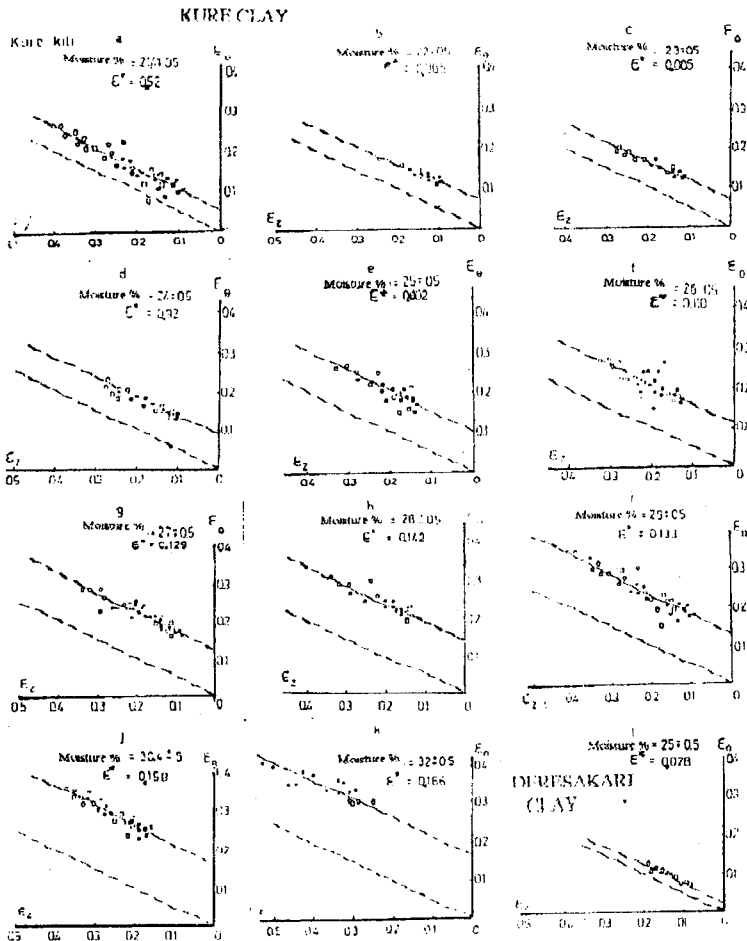
Figure 2. Forming Limit Lines for the Küre (a-k) and Deresakarı (l-r) clays.
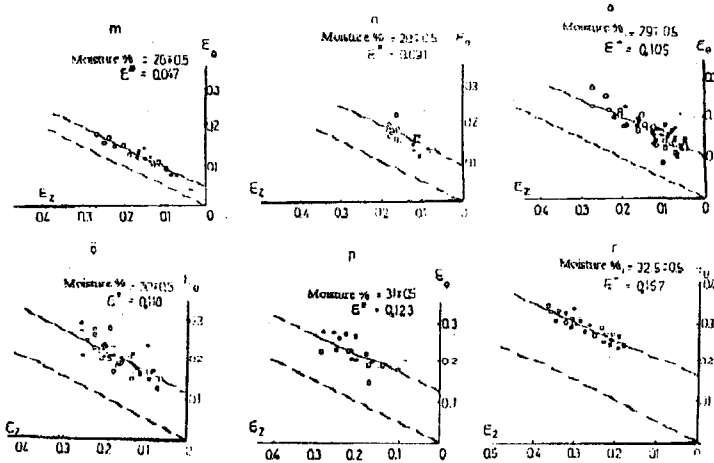
Figure 2. (Continued)

The yield limits as a function of the moisture percent, which were determined by 0.2 percent offset procedure, are given in Figure 3 for the Küre clay and in Figure 4 for the Deresakarı clay. in both clays the yield points show a sharp fall with the moisture contents. The variation of the amount of maximum deformation ($\varepsilon*$) with moisture % is given in Figure 3 for the Küre clay and in Figure 4 for the Deresakarı clay.
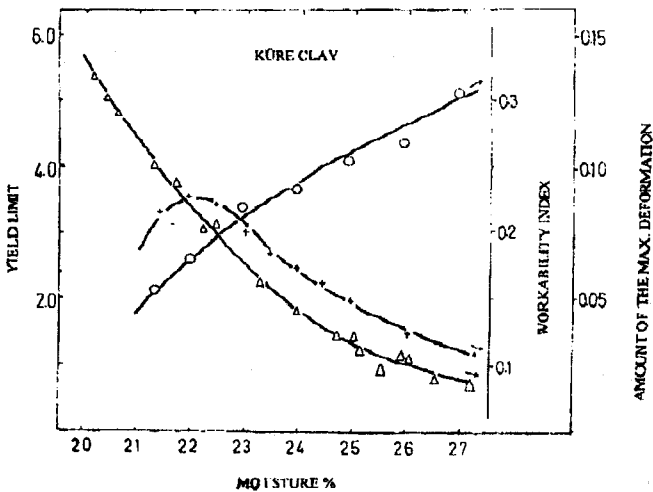

Figure 3. The variation of the yield limit (Δ), the amount of maximum deformation (o) and the workability index (+) with moisture, for the Küre clay.
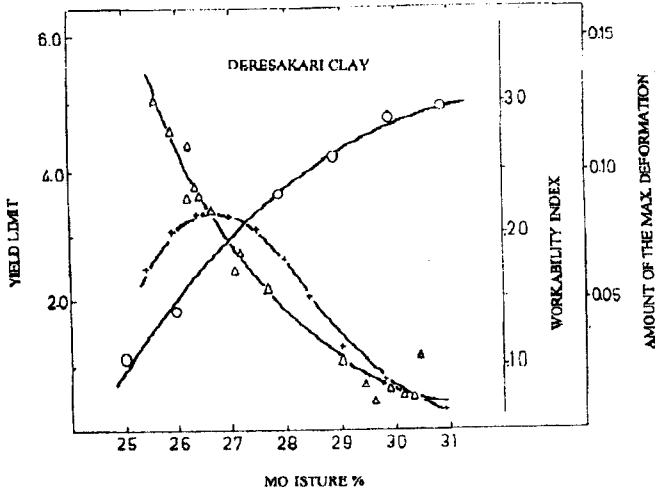
Figure 4. The variation of the yield limit (Δ), the amount of maximum deformation (o) and the workability index (+) with moisture, for the Deresakarı clay.

The product of the yield stress limit points given in Figure 1 and the amount of maximum deformation (workability indices) for the Küre clay is shown in Figure 3. Workability passes through a maximum with increasing amounts of moisture. The optimum amount of moisture for the Deresakarı clay is 22.1 % (Figure 3).

The dropping of the yield limit with the amount of moisture and the related increase in the amount of deformation for the Deresakarı clay is shown in Figure 4. The product of these two values (the workability indices) are also given in Figure 4. Here also (the workability index curve) passes through a maximum which determines the amount of optimum moisture. The amount of optimum moisture for the Deresakarı clay is 26.9%.

4. DISCUSSION

In determining the workability of clays standard experimental procedures such as tension, compression and torsion can be used. Among those, the compression experiment is the easiest one to perform. The experiments; such as tension (6), bending (7), torsion (8) and compression

(9, 10) that are used to determine the amount of maximum deformation or the special procedures developed by (3, 4) either do not measure the basic material parameters or the stress and strain positions do not represent the real procedure (3, 4, 8). In measuring the amount of maximum deformation by the torsion procedure difficulties are encountered during sample preparation and fixing the sample in the instrument. Besides those difficulties the strain positions do not resemble those of the real procedures. In the tension experiment the elongation of the sample is related to the deformation hardening, for this reason it doesn't give much information about the breaking. In addition to this, in the tension experiment the shrinking % of the area at the instant of fracture is different from the fracture at real procedures.

The procedure applied in order to determine the maximum deformation by the Norton procedure contains the strain and stress of the torsion experiment. It is a simplified form of the torsion experiment. Specially designed instruments are necessary in order to make the experiment. Besides, as it is not a standardised experiment it has not been used by other people except Kingery and Franel (11). Moreover, another of the same instrument or one that resembles this instrument doesn't exist and the original has been thrown away (12). Nevertheless, the use of this machine has been one of the most important steps towards the determination of the workability.

Workability is generally defined as the amount of deformation that can be obtained without any fracture or shrinking in a deformation process. Workability is not an internal material property, it is related to both material and process variables.

$$\text{Workability} = f_1 \text{ (process)}. f_2 \text{ (materials)} \qquad (3)$$

In expression (3) f_1 is a function of process variables such as stress, strain, strain rate and mould design and f_2 is a function of material variables such as the amount, the size the distrubution of the particles in the second phase, particle size and the amount of water % (for plastic clays). f_1 is a measure of the real toughness of the material and is represented by the workability limit criteria developed for various

processes, and f_2 is obtained from the variation of stress, strain, strain rate and temperature at the regions of possible fracture (or shrinking) of the work piece. Process variables such as the mould design, the geometry of the front piece, greasing determine the formation of stress and strain in the work piece. The path that the stress and strain follws throughout the deformation varies depending upon the process variables (f_2). Thus, in the workability expression the f_2 represents the process variables and expression (3) gives the definition of workability. The workability criteria (f_1) that give the limit stress and strain conditions are useful in determining the role of the material variables. The workability limit criteria which depend upon limit strains have practical applicability, because in the workability studies contrarily to stresses, it is easier to measure and analyse the strains.

In the mass deformation processes, free surface fractures are the ones that are observed the most. In those processes, generally the surface fractures determine the deformation limit that should be given to the material. FLL fracture criteria can be obtained from the compression and bending experiments (1). In the compression experiment, because of the friction on the mould /sample touching surface, the bulged surface which forms on the side surfaces renders harder the measurement of the yield properties of the material, whereas the most convenient region in determining the fracture strains is the equatorial region of the curved surface. In experiments made with metals under frictional conditions, tension stress in the circumferential direction and compression stress in the axial direction form on the bulged surface of the sample.
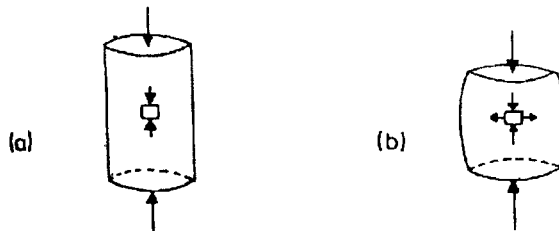

Figure 5.   Strains that form in the compression experiment under friction free and frictional
(b) conditions. Under frictional condition (b) the deformation is nonhomogeneous.

Stresses at both directions are in the form of tension under the conditions of excessive curvature. The regional strains (circumferential ε_0 and axial $-\varepsilon_z$) in the fracture region at the instant of fracture form the FLL that has a slope of -1/2 at the two dimensional coordinates. Such a line for the 1045 steel is given in Figure 6.

The spreading of the limit strains (fracture strains) in a wide interval is obtained by changing the sample geometry (height / diameter ratio) and the frictional conditions of the touching surface. Various fracture strains under a certain friction condition is obtained by changing the geometry of the sample.

During the bending of a flat metallic rod around a certain radius, stresses which resemble the stresses that occur in the bending experiment occur at the outer surface of the sample.The strains that occur at the outer surface in the bending experiment resemble the strain conditions that occur in the curved surfaces of the bending samples. As can be seen from Figure 6 the fracture strains obtained from the lines drawn to the outer surface in the bending experiment enlarges the strain interval obtained from the compression experiment. Hence, bending experiment is complementary of the compression experiment. It is useful in cases where the pressing experiment can not be made.
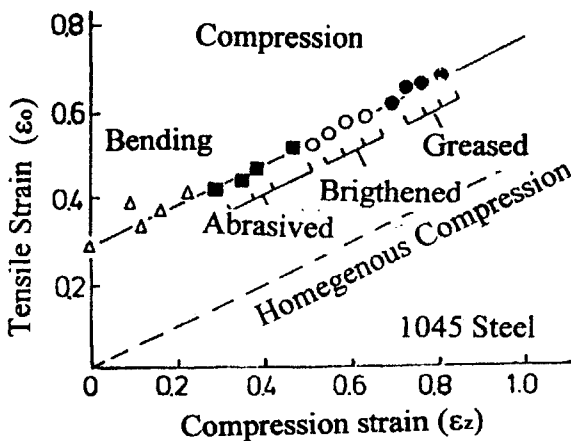

Figure 6. FLL(2) for the 1045 steel

In order to define the FLL, it is sufficient to give the intercept on the y-axis (ε^*), ($\varepsilon_0 = \varepsilon^* - 1/2\varepsilon_z$).

The height of the FLL (the intercept on the y-axis) is independent of the process variables (sample geometry, gressing etc.) and it is a material property. Hence it is an indicator of the workability of the material at a certain temperature and strain rate. The strain conditions under the FLL determine the amount of useful deformation that can be obtained from the material. The compression and tension strain conditions that are under the FLL show the permitted strain conditions (confidence area). As the height of the FLL increases the confidence area enlarges. Hence; the height of the FLL (ε^*) is an indicator of the workability of the material.

The slope of the FLL is in good correlation with the toughness breaking postulates. In this study it has been shown that the FLL (which has a slope of 1/2) relationship developed for metals holds also for the clays. As it can be seen in Figure 2 although the deviation from the data points is more when compared to the metals an exact linear relationship is observed between $\varepsilon_0 - \varepsilon_z$.

It has already been mentioned that data points could not be obtained from the compression experiments at regions where the FLL s are close to the y-axis, and that the bending experiments were used for the metals.

In order to obtain data points at those regions, the use of samples with different geometries (flanched or tapered) could be considered, but in this study it has been assumed that the fracture strains in that region would fall on the extension of the FLL and the FLL s were extrapolated to the y-axis. On the other hand, any point on the FLL that had a data point could be defined as the amount of maximum deformation without fracture. Hence; the planar strain toughness can be used as the amount of maximum deformation in determining the workability index. Besides, the definition of the planar strain toughness as the amount of maximum deformation will make the determination of the workability limits of the material under a certain amount of moisture, easier.

The strain components thus determined will be directly applicable to real processes. In the determination of the FLLs a simple frictional

condition has been used at the interface between the mould and the sample. The reason for this is that greases such as glycerine, stearic acid and vaseline can diffuse into the clay and can effect the deformation and fracture characteristics of the material. On the other hand, the FLL has been obtained by changing only the sample geometry. Hence; in this study only polished moulds and frictional conditions without grease have been used.

The coefficient of friction at the sample/mould interface has not been measured in this study. But, it can be considered that the coefficient of friction is very close to sticking conditions (μ=0.5). In several experiments made by placing a number 240 abrasive paper at the sample/mould interface, it was observed that the curving and the stress fractures were very close to those made with the same geometry without abrasive paper, so further attempts to increase the coefficient of friction was given up.

In general, the amount of maximum deformation (the height of FLLs), Figure 2 obtained from both clays is rather at a low level. In metallic materials, the planar strain toughness for the carbon steels is 0.30-0, 55, for a mono phase brass, it is 0.50-0.60 and it can increase up to 0.75 for commercially pure aluminium. On the other hand for aluminium alloys to which heat processes can be applied, the workability is low (approximetly 0.15-0.25) and is similar to values obtained from clays that were used in this study. These low levels of workability observed in clays is the result of rather low cohesion between the clay particles. For the clays, the resistance to fracture is low and this gives low workability values.

In this study the yield stresses have been obtained by using the compression experiment. In determining the stress strain curves the cylindirical clay samples have been greased with vaseline on the circular surfaces. It has been observed that vaseline diffused less in the sample when compared to glycerine and stearic acid nevertheless it was observed that as the deformation progressed vaseline diffused into the sample at a certain extent. Although a rather homogeneous deformation was obtained at the plastic region of the stress strain curves (Figure 1) it can be

thought that the plastic region has been affected from the diffusion of vaseline. Hence the deformation at the plastic region was not carried on to higher levels (approximetly 10 % strain).

Yield limits at both clays are perceptible with an abrupt passage to the plastic region (Figure 1). Although it has been observed that the plastic module decreased as the amount of water increased it can be determined by the procedures that are used. While the deformation hardening did not show much variation with the amount of moisture in the Deresakarı clay; the deformation hardening increased as the amount of moisture increased in the Küre clay (Figure 1).

The data points shown in Figures 3 and 4 show that the yield stresses decrease with the amount of moisture whereas the amount of maximum deformation increases. Hence; the product of those two values defined as the workability index forms a bell shaped curve that passes through a certain maximum. The maximum point of the workability index determined in the shape of a bell gives the product of the optimum amount of water and yield stress values. The passage of the workability curve through a maximum and the formation of a bell shaped curve is in good corelation with the results of many of the previous researchers. This shows that the use of the FLL technique in the determination of this curve is a healthy procedure. Until now many researchers have found the bell shaped curve with various procedures. The originality of the present study lies in the determination of the workability indices which are in good correlation with the findings of other researchers by the application of a simple experiment that doesn't require any special instrumentation for the determination of the amount of maximum deformation, that can be made in any laboratory and whose results are applicable to real processes.

From the plastic moisture interval the Küre clay has given higher workability levels when compared to the Deresakarı clay. The reasons for this can be stated as follows:

- The amount of SiO_2 from chemical analysis, is 63.1 % (as weight) for the Küre clay and 79.8 % for the Deresakarı clay. Besides, the

amount of SiO_2 particles (as weight) calculated by the rational analysis is 37.9 % for the Küre clay and 65.9 % for the Deresakarı clay. Free silica in the clay body is a component that effects the plasticity in a negative manner. Quartz particles do not have water adsorbent property and thus the attraction between the particles is rather low. As the yield stress is related to the attraction between the particles a low yield stress in the Deresakarı clay can be expected.

- The chemical analysis and the DTA have shown that the Küre clay contains organic matter. The effect of organic matter on the plasticity of clays is rather high. Organic matter increase the binding strength and the stickiness of clays so in most cases these are added from outside. As the Küre clay contains more organic matter compared to the Deresakarı clay it might be expected to show higher plasticity.

- The plastic fraction that passes through a 270 mesh sieve in the Küre clay is more than that of the Deresakarı clay. In the Küre clay that has particles of smaller magnitude the surface area and the adsorbtion of water will be greater and hence the plasticity will be higher.

- The X-ray diffraction shows that the Küre clay contains a certain amount of illite mineral. Illite which has a high plasticity contains rather different exehangable ions when compared to kaolinite. On the other hand the Deresakarı clay doesn't contain illite but contains quartz particles which are not plastic. Hence the Küre clay might be expected to show higher plasticity than the Deresakarı clay.

- The dry fracture resistance and the dry shrinking of the Küre clay are higher than those of the Deresakarı clay. On the other hand, the dry fracture resistance and dry shrinking are related to plasticity, and in some cases these values have been empirically thought to be an indicator of plasticity. This shows that the plasticity will be high in the Küre clay.

- The slope of the Pfefferkon plasticity water curve of the Küre clay is smaller than that of the Deresakarı clay. Besides, the slope of the curve obtained from the Plastimeter instrument is higher for the Küre clay. These show that higher workability will be obtained for the Küre clay.
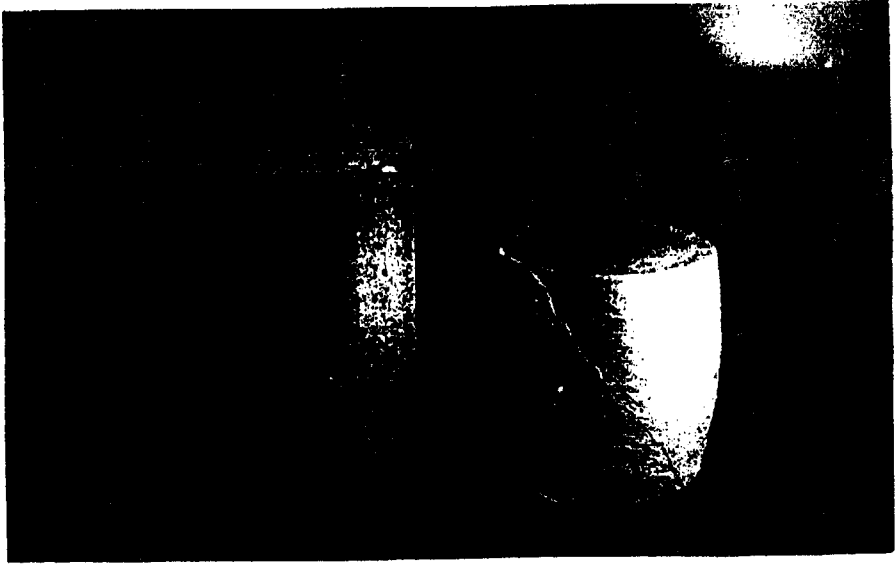
(a)
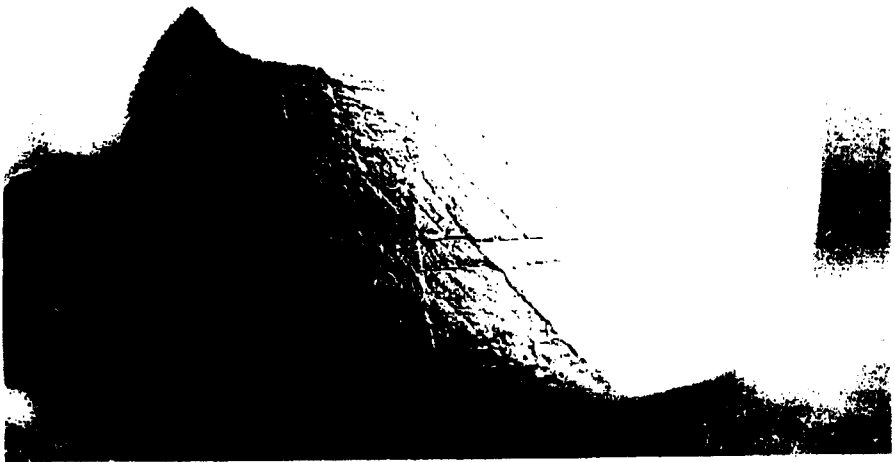
(b)

Figure 7.   The views of the fractures of 45° that occured on the samples before and after
the compression experiment.

- The Atterberg limits consist of a greater interval for the Küre clay. This also shows that the workability for the Küre clay will be greater.

- The amounts of maximum deformation obtained from the FLL technique are spread in a rather large interval for the Küre clay. This is in good correlation with the other measurement procedures for the plasticity.

5. RESULTS

1. The FLL technique, that relies on the pressing experiment under frictional conditions which is successfully applied for the workability of metals can also be used for the determination of the limit fracture strains in the clays.

2. By multiplying the amounts of maximum deformation obtained from the FLL technique by the yield stresses obtained from the compression experiments the curve of the workability indicator with respect to the amount of moisture % can be obtained. This curve passes through a maximum that determines the optimum amount of water. Hence a new procedure for the determination of the workability of clays, which relies completely on the compression experiment and which is based on the maximum deformation without fracture and also on the yield stress, has been developed.

3. The Küre clay has given a higher level of workability with respect to the Deresakarı clay. The reasons are that the amount of SiO_2 in the Deresakarı clay is higher, the Küre clay contains organic matter, the fraction which passes through a -270 mesh sieve of the Küre clay is larger and the Küre clay contains illite mineral that is plastic.

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