THE CHARACTERIZATION OF PLASTICITY AND OBSERVATIONS ON AGING

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Abstract

The plasticity of a ceramic forming body is critical to controlling the fabrication process. A new technique has been developed to quantitatively measure plasticity using high pressure shear rheometry. The results from the plasticity measurements have also been correlated with plastic forming processes. This data will be presented and observations on the aging of clay-based systems will also be discussed.

Introduction

Plasticity is undoubtedly one of the most poorly understood properties of a clay body. It could be argued that there are two types of plasticity: good and bad. If a material works within a specific process, the plasticity is perceived as good. If the material does not function, or creates defects in the product during the forming process, the plasticity must be bad. The major problem lies in the fact that plastic masses are frictional solids, placing them outside of the suspension rheology concepts (for fluid materials), and thus closer to the range of materials addressed by soil mechanics.

In evaluation of material remaining in a extrusion mill, lead consistently to the conclusion that the material failed in shear under considerable pressure during the forming process. The pressure for extrusion was provided by the crew, and that material underwent shear-type failure in order to extrude. This analysis provided an alternative approach for the chracterization of plasticity, one which required that the shear behavior be measured under pressure. In addition, because it appeared to be necessary fot the samples to be tested at elevated pressures, unconfined testing procedures (i.e., the Atterburg of Pferfferkorn tests, as well as torsion or mechanical tests such as those wich measure the resistance to flow) would not be applicable. Other testing procedures, such as evaluating the extrusion of samples. would be limited by whether or not the samples could be extruded - no extrusion, no data. A critical review of plasticity testing is currently awaiting publication. [1]

In order to better quantify the plasticity in ceramic particle systems, a new technique has been developed using high pressure shear rheometry. This technique allows the shear behavior of samples to be measured as a function of pressure, independent of the forming process. The range of operation of the material can be determied by evaluationg the test samples with the fabrication technique for which the material is intended. One of the primary advantages of this technique is that it is independent of the fabrication process, and therefore does not require the sample to actually be useable in order to be tested.

Concepts of the testing approach

Design of the high pressure shear rheometer

The high pressure shear rheometer, referred to as the High Pressure Annular Shear Cell, or HPASC, is illustrated schematically in Figure 1. The instrument consists of an annular testing chamber (Figure 2a) a hydraulic loading apparatus, and a variable rotation system. Measurements are taken electronically and collected via an electronic interface in a spreadsheet. The confining and compaction pressure is applied via a hydraulic pressure amplifier, in the form of a lever arm and weights, as pictured in Figure 2b. Because the instrument is still under evaluation, the testing procedure takes between one and two hours to complete.

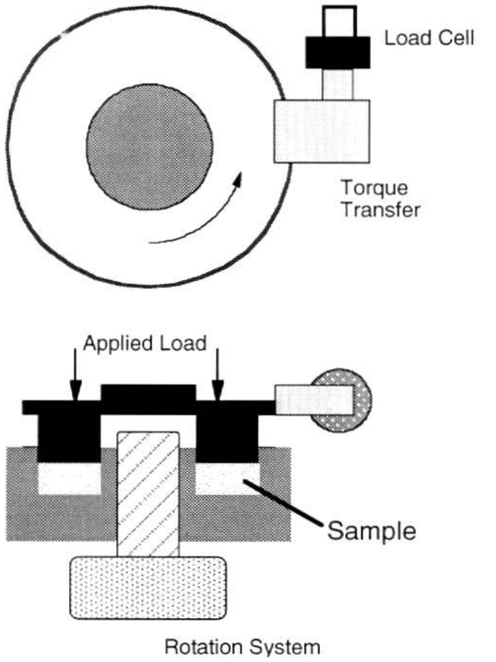
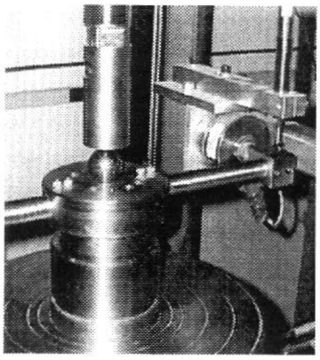


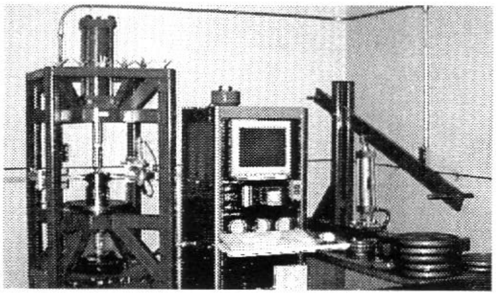
Figure 1. Schematic illustration of the High Pressure Annular Shear Cell.

The underlying principle for the characterization of plasticity using the HPASC stems from the concept of a «Bingham-type» fluid, which is a Newtonian-like fluid with a yield stress (Figure 3). Since plastic masses are granular masses, (also referred to as frictional solids) their behavior is highly pressure dependent and thus the Bingham yield stress will increase with increasing pressure, as illustrated in Figure 4. From this perspective, it is easy to recognize that the pressure dependence of samples changes as a function of water content, as illustrated in Figure 5. In the wet case, water fills the pores (i.e., the system is saturated) and increasing pressure has little effect on the shear strength of the mass. Since water is incompressible, an isostatic pressure case results and there is little change in the particle-particle interactions. In the dry case, a small increase in pressure results in a large increase in the shear strength due to a corresponding increase in the friction between the particles. Somewhere between these two extremes lies plastic behavior. In addition, it is evident from Figure 5 that there is infinite range of possible behaviors.

The yield stress values, when plotted as a function of pressure (illustrated in Figure 6a), reduce the data to a usable form, and provide a quantitative characterization of plasticity. The slope (representing the pressure dependence, or sensitivity of the sample shear strength



a) EMBED Word.Picture.6



b)
Figure 2: (a) A photograph of the HPASC testing fixture with the testing chamber as illustrated schematically in Figure 1. (b) Photographs of the three components of HPASC instrument: (from left to right) (i) the HPASC testing frame, (ii) data collection unit and computer terminal, and (iii) dead weight loading arm system with hydraulic amplifier.

to the applied pressure) and the intercept (or cohesion, representing a critical shear stress for zero shear rate and zero pressure) are calculated from the data and used as the two parameters of plasticity. The intercept (on the y-axis) is then plotted as a function of the slope (on the x-axis) to create a two dimensional surface (Figure 6b) which can be used to

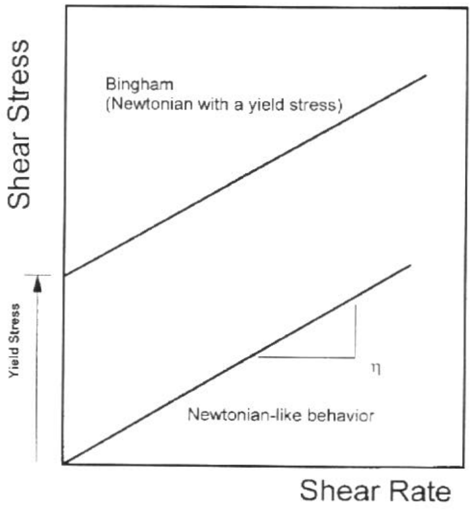


Figure 3. Schematic illustration of Newtonian-like and Bingham fluid behaviors.

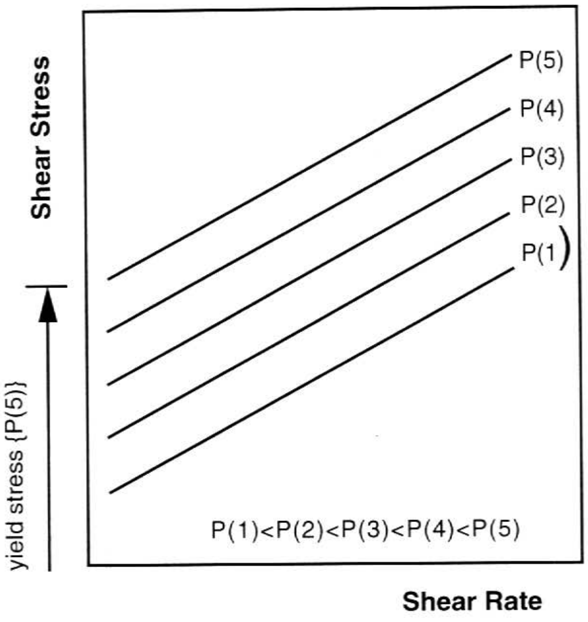


Figure 4. Schematic illustration of the effect of pressure on a Bingham fluid.

describe a broad range of plastic behaviors. Using the process (such as extrusion) to screen the samples, the range of plasticity necessary for that process can then be defined, as represented by the region marked «extrudable range.» (Figure 6b is only a cartoon of the concept — this region must be defined and may not necessarily follow this shape.)

To move around on the plasticity diagram requires changes in the slope and intercept.

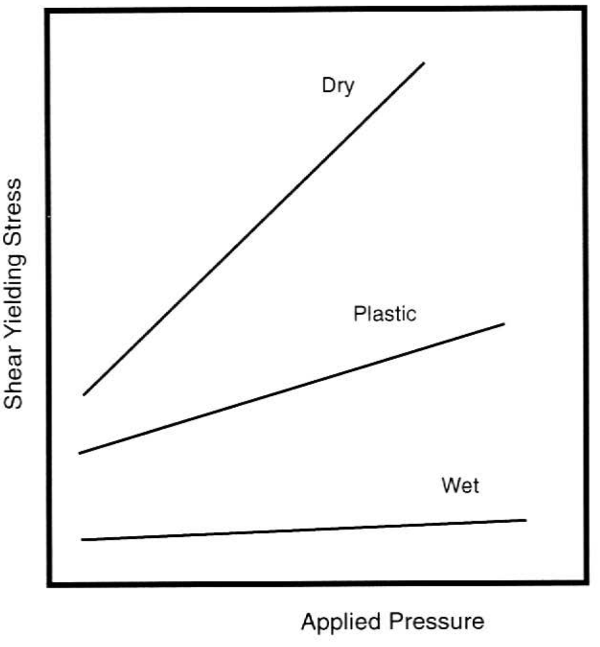


Figure 5. Cartoon depicting the range of plastic behaviors.

Figure 7 illustrates that samples A and B have identical intercepts but different slopes, causing them to be located on a horizontal line in Figure 7b. Samples B and C have the same slope, but different intercepts, locating these samples on a vertical line in the plasticity diagram (8b).

Experimental Approach

Testing procedure

Between 75 and 100 grams of sample are loaded into the test fixture. The load is first applied as the sample is rotating at the slowest rotation rate. To allow the sample to achieve steady-state, and thus ensure repeatability, the sample is allowed to equilibrate at least two complete revolutions before data is collected for a measurement. After the data point is collected, the pressure is increased (or the rotation rate is changed — depending on the desires of the operator), and the equilibration/ data collection process is repeated. The range of rotations rates is from 0.3 RPM to 2.5 RPM. The range of testing pressures is from 5 MPa to 100 MPa (although most testing is conducted between 10 and 40 MPa).

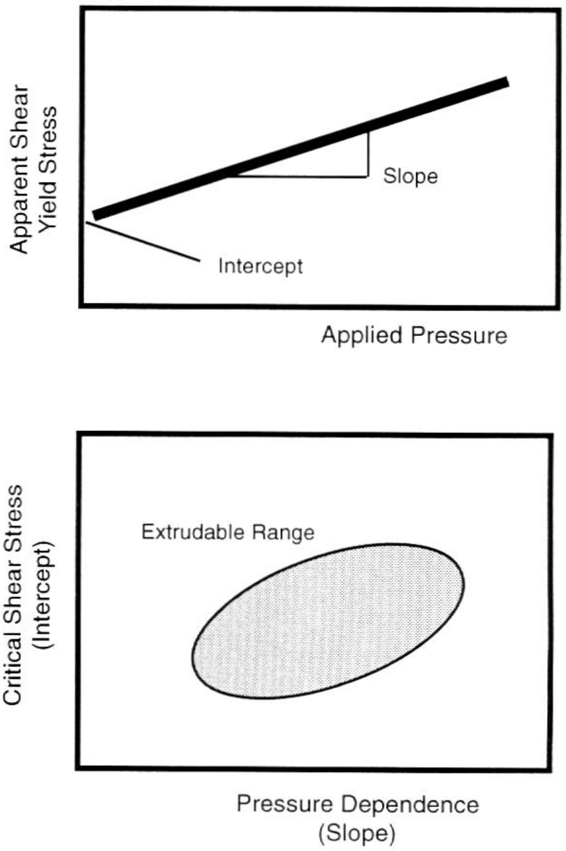


Figure 6. The definition of the slope (the pressure dependence) and the intercept (the critical shear stress). The bottom figure shows how the slope and intercept are then used to create the plasticity diagram with the extrudable range defined by screening the samples using the process tool.

Sample preparation

A variety of samples were prepared using a mix-muller for mixing. Other samples have been prepared using a slurry approach method, followed by filter pressing. Some samples have also been prepared via spray drying, but only the data from the mix-mulling samples will be presented at this time.

The initial studies consisted of the testing of a commercially available (in the United States) kaolin and ball clay (a primary clay consisting of almost pure kaolinite, and a secondary clay with the major impurity being quartz, respectively). The clay samples were mixed with de-ionized water, a sample removed, and additional water added, to provide a

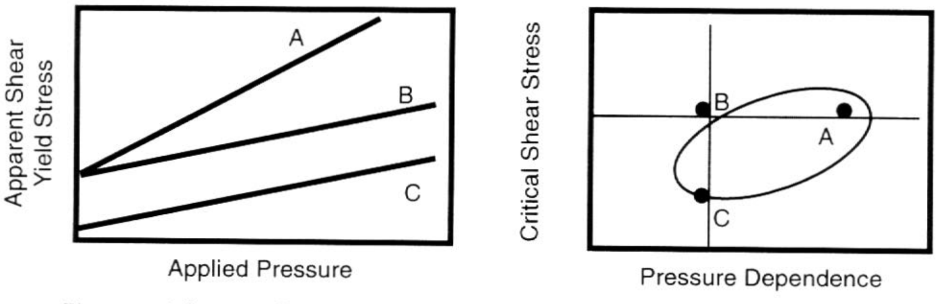


Figure 7. Schematic illustration depicting how changes in plasticity alter the location on the plasticity diagram.

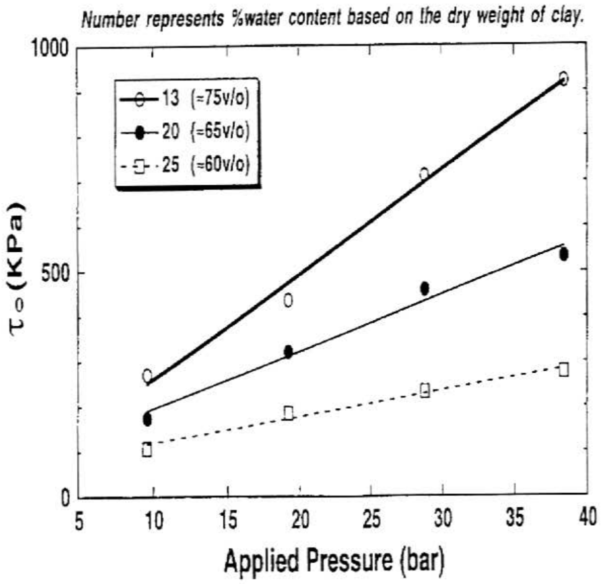


Figure 8. Yield stress versus pressure for three ball clay-water mixtures.

range of samples with varying water contents. Knowing that the water content can dramatically alter the properties of a clay-water mixture, this procedure allowed the trends and contribution of water to a clay sample to be evaluated without having to worry about achieving or maintaining exactly the same water contents. Samples were dried prior to testing to determine the exact water content of the test specimens.

Samples for aging were prepared, marked, and sealed in air-tight containers. Multiple containers were used to eliminate variations introduced by opening the sample containers to withdrawn samples.

To evaluate the contribution of non-plastics (i.e., feldspar and quartz) to the plasticity, a standard batch was also prepared in an identical fashion using the mix-muller.

Results and Discussion

Figure 8 shows the yield stress versus applied pressure data for samples of ball clay containing three water contents. These represent examples of wet, plastic, and dry, demonstrating that the concept appears to be valid and that the data strongly reflects the cartoon illustration depicted in Figure 5. It should also be noted the extremely high degree of linearity in the data, helping to keep the evaluations objective.

Figure 9 shows the variation between plasticity obtained from kaolin, ball clay, a mixture of two parts ball clay to one part kaolin, and to the standard triaxial porcelain batch. This plot demonstrates several interesting results. First, it is clear that differences between secondary clays and primary clays are easily discernible and can even be distinguished from a mixture of the two clays. Second, the water content necessary to reach a maximum cohesion is highest for the secondary clay, lowest for the primary clay, falls in between the two for the mixture. This value should represent the saturation of the body with water, and may reflect the water of plasticity necessary to provide plasticity to the material. In addition, the water content required for maximum cohesion should correspond to the specific surface areas for the clays and as illustrated

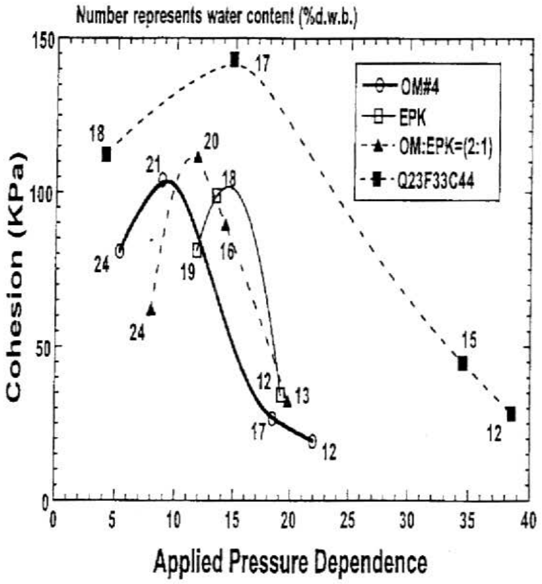


Figure 9. Plasticity of ball clay (OM#4), kaolin (EPK), a 2:1 mixture (ball clay:kaolin), and standard porcelain batch (23% quartz, 33% feldspar, and 44% ball clay:kaolin mixture) showing the dependence of plasticity on water content (denoted by the values on the plot next to data points).

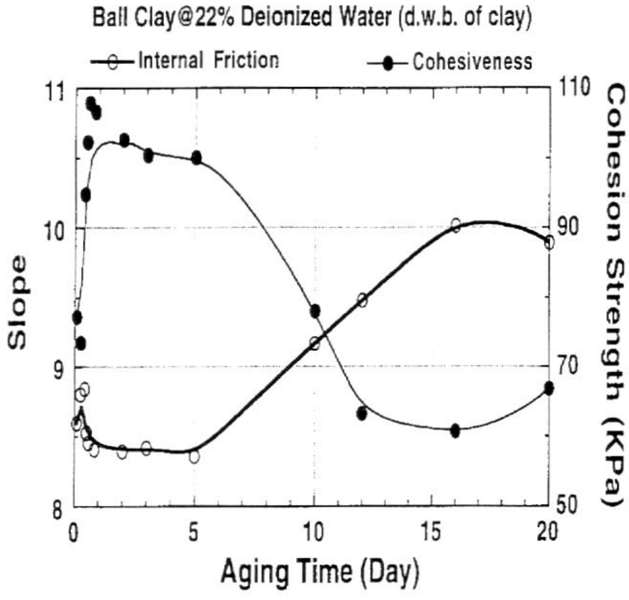


Figure 10. Plasticity as a function of time for mixture of ball-clay and 22% de-ionized water (on a dry weight basis).

in Table I, the ball clay has a higher specific surface area compared to the kaolin. It should also be noted that the pressure dependence decreases with water content as is predicted by the overall concept and that as the saturation point is exceeded, the pressure dependence becomes more sensitive to water content.

When evaluating the contribution of the non-plastics, it is evident that significantly higher cohesion stresses are obtained at lower water contents than required by a clay-water mixture by itself. Two factors are at work: (i) the specific surface area of the mixture is significantly reduced by the substitution of the coarser particle size non-plastic fractions; and (ii) the particle size distribution of the body has been dramatically altered by the introduction of the non-plastics. With regards to the second point, the introduction of larger particle size material significantly improves the packing behavior of the body, resulting in increased particle-particle contacts, and greater cohesion stresses. The reduction in surface area results in less water being necessary before the body saturates.

Figures 10 and 11 depict the change in plasticity of a clay-water mixture and of the standard composition, respectively, as a function of time. Both of these samples were prepared without the use of any dispersants or surfactants and represent the flocculated state of the samples in de-ionized water. It is evident from the data that the properties of the samples change dramatically with time and that the most substantial change occurs in the first day or several hours. In addition, it is apparent that the non-plastic additions accelerate the aging process, requiring significantly less time to reach a steady-state condition than that required for the clay mixture by itself.

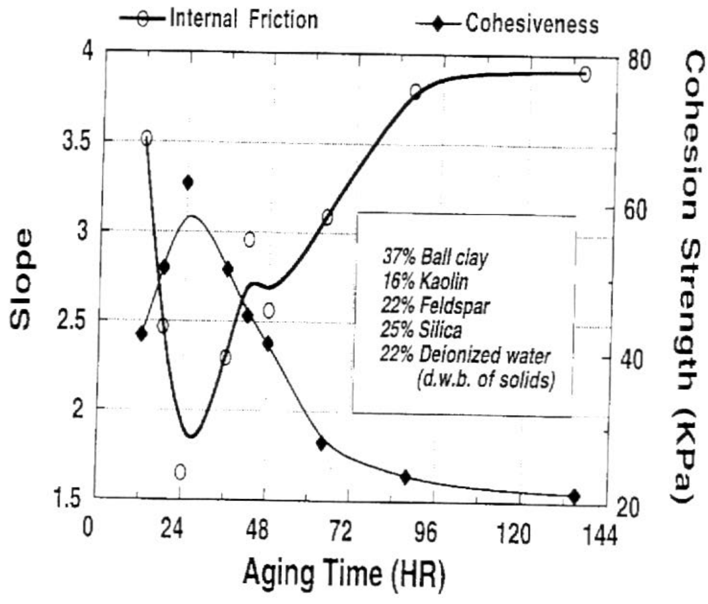


Figure 11. Plasticity as a function of time the standard porcelain batch (23% quartz, 33% feldspar, and 44% ball clay:kaolin mixture). (Note: Slope = internal friction; and Cohesion strength = cohesiveness).

Summary and Conclusions

A new technique for evaluating plasticity has been developed which is independent of the forming process. This technique, using high pressure shear rheometry, allows plasticity to be quantitatively evaluated and characterized. From the results presented, it was possible to demonstrate the differences between a primary clay and a secondary clay, to distinguish between mixtures of the two, and to identify the water content necessary for the highest cohesion. In addition, the role of the non-plastic additions (feldspar and quartz) in the plasticity of a clay body to be measured, showing that significantly lower water contents are required to obtain high cohesive stresses. Finally, the impact of time on the plasticity of a clay-based system has been, for the first time, quantitatively determined.

References

1. Nowak, P, and Carty, W., «A Critical Review of Plasticity Characterization,» Proceedings of the Science Technology and Commercialization of Powder Synthesis and Shape Forming Processes Symposium, #SXIX, Cincinnati, OH, 1994, American Ceramic Society, Westerville, OH, (awaiting publication).