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Rheometric and ultrasonic investigations of viscoelastic properties of fresh Portland cement pastes

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Abstract

The paper investigates the possibility of using a shear wave reflection technique to monitor the viscoelastic behavior (represented by storage shear modulus and viscosity) of Portland cement paste at very early age. Three cement pastes with water/cement ratios equal to 0.4, 0.5 and 0.6 cured under water at a constant temperature of 25 °C were studied. By measuring the wave reflection coefficients and the phase angles of reflected ultrasonic waves, the dynamic storage shear moduli and the viscosity of the cement paste can be calculated. The calculated results of the storage modulus were compared with the results obtained directly from the oscillatory rheometric measurement. In addition, the viscosity calculated from the wave reflection measurements was compared with results obtained directly from the step rheometric method and a qualitative agreement was found. The results show that as a non-destructive method, the ultrasonic wave reflection method provides useful information about both the elastic and viscous behavior of cement pastes at very early age.

Keywords: Viscoelastic behavior; Ultrasonic wave reflection coefficient; Oscillatory rheology; Storage modulus; Viscosity

1. Introduction

When cement paste is subjected to an external force, it responds in a manner intermediate between an elastic material and a viscous material. This viscoelastic behavior changes during the hydration process of the cement particles [1]. A better understanding of the viscoelastic properties of cementitious materials can help to control the volume stability of the material at later ages.

Viscoelastic properties are important not only for cement-based materials at later ages; it is also a factor that affects the workability of the material in the very early age. The knowledge of viscoelastic properties can provide fundamental information about the physical status of a solid particle suspension system transforming to a viscous semi-solid flowable and subsequently to a solid system.

This study will mainly focus on the viscoelastic properties of Portland cement pastes at very early age, namely before the initial setting time. The methods used in the research to investigate the viscoelastic properties of the cement pastes at early age are based on the theory of dynamic rheology [2].

In 1950s, Mason [3] introduced the concept of non-invasive ultrasound viscometry. He measured ultrasonic wave reflections from a liquid–quartz interface and used this data to determine the viscosity response of the fluid. Since then, ultrasonic shear waves have been used to characterize the viscosity of the fluid in several ways. Parameters such as wave amplitude, phase angle and the frequency of the wave were used to monitor the viscous or viscoelastic properties of thick fluids [4–7]. By using shear waves, the measurements can be correlated to viscosity, which is the parameter of interest for most engineers and researchers.

The ultrasonic wave reflection technique was first applied to the area of cementitious materials by Stepišnik et al. in 1981 [8]. This method can be used to monitor the setting and hardening behavior [9] and can also be adopted to mimic the strength development of cementitious materials at early age [10–13]. In a recent study, more attention was paid to the application of this method to monitor the viscoelastic properties of cement pastes [14]. It was shown that when cement paste is in solid state, the

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material behavior can be approximated by neglecting the phase shift measured by the wave reflection technique [8,15]. But few results on fresh cement paste in flowable status were reported.

In this study, the application of the ultrasonic shear wave reflection technique was explored. The technique was used to monitor the viscoelastic properties of cement pastes at very early age by observing the evolution of the storage shear modulus and the viscosity. The calculated storage shear modulus, which represents the elastic properties of the materials were compared to the results measured directly from the oscillatory rheometric method, which is from a micro-structural point of view also a non-destructive testing method. In this oscillating rheometric method [16–18], the storage and loss shear moduli of the cement paste can directly be measured by applying oscillating shear strain according to a sine function and measuring the corresponding shear stress. By controlling the value of oscillatory shear strain and the frequency within the linear viscoelastic region of the material, the microstructure of the cement paste will not be destroyed during the oscillations, and the evolution of the material properties during hydration can be observed.

2. Theory background

2.1. Material properties

If mechanical energy acting on a body is partly stored in deformation and partly dissipated in other forms, then such behavior is defined as viscoelastic behavior [19]. Concrete is a typical viscoelastic material. The porous structure of the paste matrix and the interfacial transition zone between cement paste and aggregates make the properties of concrete more complex. Understanding the viscoelastic properties of Portland cement paste can be helpful to better understand the behavior of concrete.

The deformation of a viscoelastic material is not proportional to the applied force because of the energy dissipation. In this context, dynamic methods can help to distinguish between the elastic and viscous properties of the material. When a linear viscoelastic body is subjected to stress varying sinusoidally with time at a certain frequency, the corresponding strain is not in the same phase as applied stress, which results in a phase lag between strain and stress as shown in Fig. 1(a) [19]. The applied stress can be separated into two independent components: one is exactly in phase with strain, the other is $\pi/2$ out of phase as shown in Fig. 1(b) [19].

The viscosity, which represents the resistance of the fluid to flow, can also be calculated through the wave reflection technique. The calculated results were compared to the viscosity measured directly from the step rheometric method [20]. In the step rheometric method, the strain rate varied within a certain range in uniformly changing steps and the corresponding stresses at each strain rate step were measured. The comparison of the viscosities obtained with the two methods is discussed in a later part of this paper.

According to the decomposed stress components, the relationship between stress and strain of a viscoelastic material can be established by using the modulus of rigidity in a complex format. If a material is subjected to a shear deformation, the shear modulus can be expressed as follows,

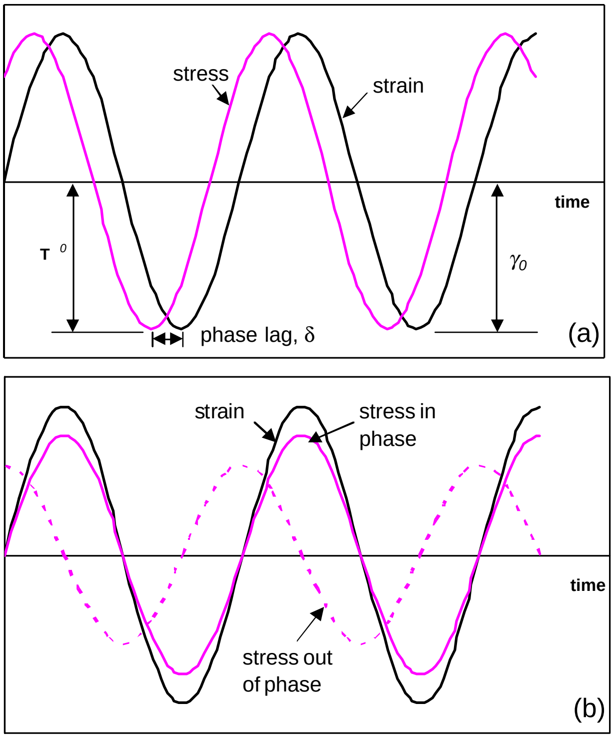

Fig. 1. Stress and strain relation in a viscoelastic material [3].

$$G^* = \frac{\tau}{\gamma} = G' + iG'' \tag{1}$$

$$G' = \frac{\tau_0}{\gamma_0}\cos\delta \tag{2}$$

$$G'' = \frac{\tau_0}{\gamma_0}\sin\delta = \omega\eta' \tag{3}$$

where G^* is the complex shear modulus, G' is the storage shear modulus, which represents the elastic behavior or the energy storage of the material, and G'' is the loss shear modulus, which represents the viscous behavior or energy dissipation of the material. If the physical status of the viscoelastic material is in flowable status, the loss shear modulus can also be related to the in-phase viscosity of the material [8,14,15] as shown in the second part of Eq. (3), where ω is the angular frequency of the applied oscillating strain (stress). The phase lag(δ) between stress and strain, which changes between 0 and $\pi/2$ can also be used to describe the material behavior. When a material is ideally elastic, the phase lagδ equals to zero; when a material is ideally viscous, parameter δ equals to $\pi/2$.

2.2. Linear viscoelastic region (LVER)

Generally speaking, the elastic moduli of a viscoelastic material are time dependent. However, there is a specific region, called linear viscoelastic region (LVER) [21], under which the elastic moduli of such a viscoelastic material are