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8. An experimental analysis of the determination of the elastic modulus of cementitious materials

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Abstract: The paper is focused on experimental analysis of cementitious materials. The determination of the elastic modulus is of special interest. The experiment results showed the elastic modulus of cementitious materials can be determined during initial stages when the internal microstructure is still beginning to form.

Keywords: elastic modulus, cementitious materials, experimental analysis

8.1. Introduction

Over its fairly short existence, concrete has become one of the most commonly used building materials (Abdelgader *et al.*, 2014, Aïtcin 1998). The rapid development in this area had the effect that the concrete of today must meet far stricter requirements than the concrete of several decades ago (Tsai and Hsu, 2002). Unlike the past, compressive strength is no longer the only important property (Cikrle and Bílek, 2010); other characteristics have been the subject of attention of engineers and researchers and have been extensively measured and discussed. Aside from the commonly examined parameters, such as durability, shrinkage, or sustainability and environmental impact, these are mainly deformation properties, the most notable of which is the modulus of elasticity (Tang *et al.* 2015, Collepardi, 2010). Development of new types of concrete, e.g., self-compacting (SCC), high-strength (HSC), ultra-high-performance (UHPC), or freshly compressed concrete (FCC) (Nematzadeh and Naghipour 2012), has been shifting the boundaries of material characteristics and concrete properties (Dehestani *et al.* 2014, Ma *et al.* 2016, Procházka *et al.* 2011, Parra *et al.* 2011). The use of various modern admixtures and additives can significantly improve the compressive strength of concrete (often exceeding 200 N/mm^2), however, their effect may not be as strong in terms of increasing the modulus of elasticity (Gu *et al.* 2015).

The modulus of elasticity is one of the most important physical properties that characterise a material. Concrete is undoubtedly one such material, especially when it comes to structural calculations. The elastic modulus is an important parameter for calculating deflections and creep especially in long-span elements and in pre-stressed or post-tensioned structures (Navrátil 2008, Neville and Brooks 2010, CEB-FIP Model Code 1990, 1993). Besides deformation calculations, in structurally uncertain buildings it also enters the calculation of internal forces; it thus affects the determination of the limit states of the load-bearing capacity. The elastic modulus is indicative of the deformation behaviour of concrete structures, such as deflections, shifts, shrinkage, or elevation in pre-stressed elements (Ma *et al.* 2016). This is why it has recently seen extensive investigation by a rising number of experts, companies, and institutions (Křížová *et al.* 2013, Huňka and Kolísko 2011).

8.2. Theoretical background

In order to properly explain the concept of the elastic modulus, it is necessary to start in broader terms, i.e. the theory of elasticity and plasticity. The two main points of examination in the theory of elasticity are stress and strain. The theories of elasticity and plasticity draw on fields that focus on studying the strength of materials, which is the ability of a material to resist external forces without sustaining damage. In civil engineering, elasticity and plasticity constitute the foundation for the theory of structures.

In elastic materials there is a clear dependence between stress and strain caused by stress, which applies in all stages of its action, i.e. during loading, unloading, loading in the opposite direction, etc. In an elementary body stressed along the x axis there is a linear relationship between strain ε_x and normal stress σ_x expressed by Hooke's law as:

$$\sigma_x = E \cdot \varepsilon_x \tag{8.1}$$

Where:

σ_x – stress in N/mm^2,

E – modulus of elasticity (also Young's modulus) in N/mm^2,

ε_x – strain in [-].

A precise relationship between changes in the shape and dimensions of bodies and the loading of a real material by external forces can never be precisely determined in advance, because it depends on many factors, mainly the properties of the material. It cannot even be derived from the physical nature of the material being tested. However, when designing building structures or elements, and especially when determining their deformation, the knowledge of the calculation characteristics corresponding to this dependence is necessary. The relationship between the deformation of solid bodies caused by external forces can be plotted as an idealised *F-d* curve, or, after conversion, as a σ-ε curve, see e.g. (Neville 2011). In real materials, the curves are substantially more complicated. However, the records of their real progress can usually be approximated and replaced part by part by the ideal curves (Navrátil 2008, Neville 2011).

The real behaviour concrete is more complex. Several types of the compressive modulus of elasticity can be identified depending on which part of the stress-strain curve is considered and what type of line (tangent or secant) is fitted (Newman and Choo 2003, Neville 2011, Navrátil 2008). The initial modulus of elasticity is identified by the standard (EN 1992-1-1 2004 and Navrátil 2008) as E_c, the other standard (EN 12390-13 2013) $E_{c,0}$ and in (Huňka 2014) it is E_0. This is a modulus of elasticity that concrete should exhibit at very low stress. According to the standard (EN 1992-1-1 2004) its value can be considered 1.05 times of the secant modulus of elasticity E_{cm}. The value of the static modulus of elasticity commonly considered for ordinary concrete ranges between 20 and 40 GPa (Neville 2011). On the other hand, the dynamic modulus of elasticity reaches higher values, ranging from 30 to 50 GPa for ordinary concrete. The determination of the dynamic value operates with the very beginning of the stress-strain curve, because the specimen suffers virtually no strain. The dynamic modulus can thus be considered tangent (Neville 2011). However, it is different from the initial (also tangent) modulus of elasticity given the way it is defined in (EN 1992-1-1 2004, EN 12390-13 2013). The ratio between the static and dynamic values of the elastic modulus is substantially higher than 1.05 as written therein.

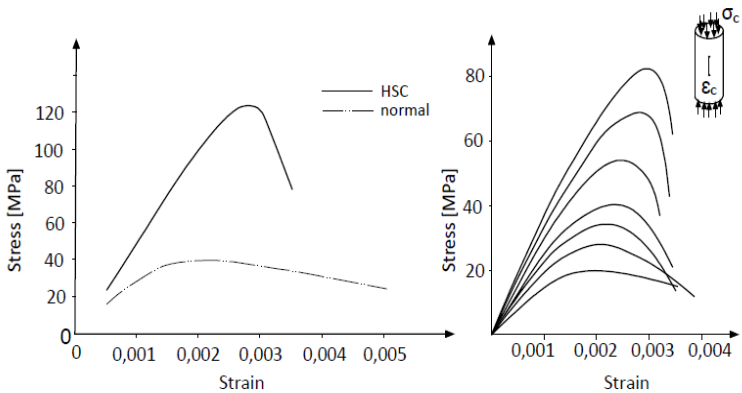

Fig. 8.1. Idealised comparison of stress-strain curves for high-strength and ordinary concrete in compression (Newman and Choo 2009) (left) and real stress-strain curves for concrete in compression (Nilson and Winter 1991) (right)

It must be understood that, unlike e.g. steel, the elastic modulus of concrete is not one specific number, but can vary due to a number of reasons, and in some situations this variation may be rather significant. It is also given by the way the stress-stress-strain curves are plotted (the slope of the first part of the diagram) for concretes of varying strength. Fig. 8.1 compares stress-strain curves from different authors.

The elastic modulus of concrete in compression (tension) depends mainly on its composition, which affects all of its properties (CEB-FIP Model Code 1990, 1993, Yildirin and Sengul 2010, Hela and Křížová 2011). Still, it would be wrong to say that composition is the only factor. There are a great number of factors that affect the final value of the elastic modulus (Lydon and Iacovou 1995). These can be divided into technological and testing-related ones (Huňka *et al.* 2013). Composition is undoubtedly a technological factor. The elastic modulus will be affected mainly by the type of aggregate (Mitrenga 2011, Aïtcin 1998), but also by the content of admixtures, especially air-entraining (Vymazal *et al.* 2011), various other additives and their amount (Křížová *et al.* 2013), or the w/c ratio (Newman and Choo 2003, Neville and Brooks 2010). Another one of these factors can also be the curing conditions present during early stages of setting and hardening. The ambient temperature is the most decisive factor in that respect. Curing especially in difficult conditions – in winter (concrete must be heated during construction at temperatures below freezing), or in summertime (concrete must be protected from desiccation at high temperatures) – is an important factor that influences the elastic modulus in no small way (Kocáb *et al.* 2017). Fig. 8.2 – 8.5 display examples of the technological factors influencing the value of the elastic modulus.

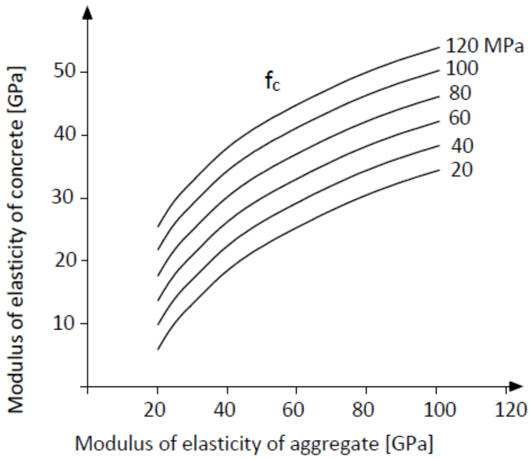

Fig. 8.2. Nomogram of the dependence of the elastic modulus of concrete on the elastic modulus of aggregate and compressive strength (Aïtcin 1998)

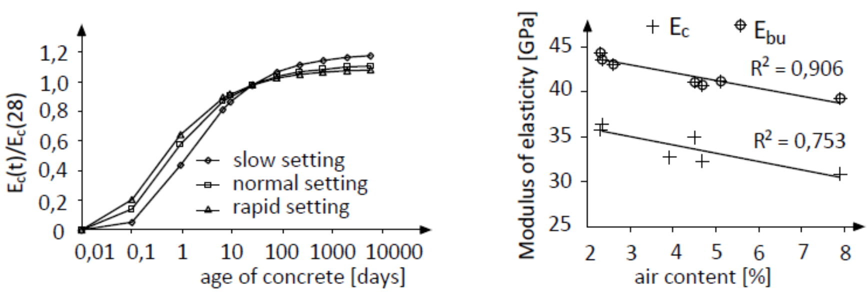

Fig. 8.3. Development of the modulus of elasticity (CEB-FIP Model Code 1990, 1993); the x axis has a logarithmic scale (Navrátil 2008) (left); plot of the dependence of the static (E_c) and dynamic modulus of elasticity (E_{bu}) on air content (Vymazal *et al.* 2011) (right)

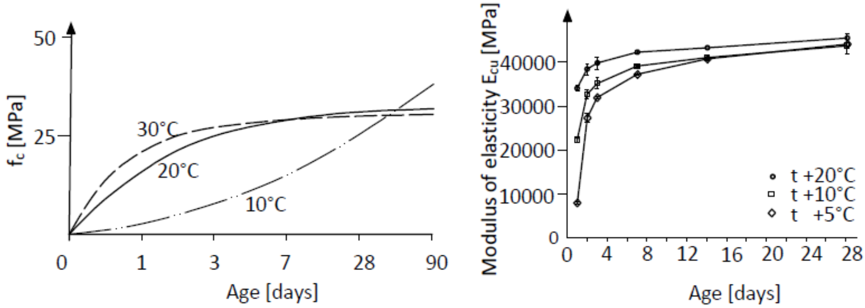

Fig. 8.4. Influence of the ambient temperature on the development of compressive strength (Collepardi 2010) (left) and on the development of the dynamic modulus of elasticity (Kocáb *et al.* 2017) (right)

Another category of factors influencing the value of the elastic modulus are testing-related factors. This category includes, e.g., the choice of method to determine the elastic modulus, what specimens are used, or how the ends of the specimens are finished (Huňka 2014). Technological factors can be seen as real factors, because, for instance, using a different aggregate results in a real change in the value of the modulus of elasticity. On the other hand, the testing-related factors can be seen as largely "apparent". While a measurement of the modulus of elasticity performed on a cylinder will return a different value than measurement made on a prism, the real value of the modulus of elasticity for that particular concrete remains the same.

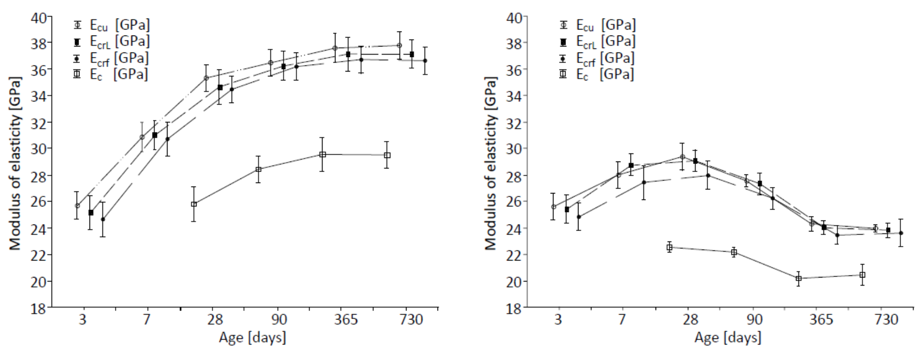

Fig. 8.5. Diagram of the progress of the average values of elastic moduli for concrete; a) set of cured specimens and b) set of uncured specimens (Kocáb *et al.* 2017)

If we do try to categorise testing factors, we can establish two basic groups - those that can be influenced (e.g., specimen preparation, loading rate, etc.) and those that cannot (method for determining the dynamic modulus vs. the static modulus of elasticity). One must also realise that the modulus of elasticity measured on a specimen will never quite correspond to the real modulus of concrete used in a structure – the concrete in the specimens ages differently than concrete in a structure. If the modulus of elasticity is measured on a structure, one is faced with limitations of the testing methods or equations for calculation.

There are multiple methods that can be used to determine the value of the elastic modulus of concrete. A basic division can be the method of measuring the elastic modulus; static methods make use of introducing load to the specimen while measuring strain, while dynamic methods usually operate on non-destructive electroacoustic principles and use the physical law for flexural wave propagation in solids (Huňka 2014). In general, the value of the dynamic modulus of elasticity tends to be higher than the value of the static modulus. The difference between the static and dynamic value depends mostly on the quality and age of the concrete. A general rule is that the lower the quality of the concrete (or the younger the concrete), the smaller the ratio between the static and dynamic modulus (can be as low 0.6). On the other hand, in very high-quality concretes this ratio can be relatively high (approx. 0.9). It is thus clear that measurements e.g. by the resonance method, which returns the dynamic (tangent) modulus of elasticity, the result will be a different number than using, e.g., the compressive strength test, which returns the static (secant) modulus of elasticity. Moreover, neither static methods (mostly compression and flexural load) nor dynamic methods (ultrasonic pulse velocity, resonance, or the impact-echo test) give the same result. Another factor is the shape and size of specimens and also the quality and a flatness of the surfaces which are in direct contact witch the plates of testing machine.

The testing of the modulus of elasticity carries with it a multitude of other factors that influence the modulus of elasticity, although mostly only in a small way – e.g. the material and dimensional accuracy of the moulds used to make the specimens, the type of instrument used (e.g. ultrasonic tester or strain gauge), or the loading rate during the compressive strength test when determining the static modulus of elasticity (Huňka *et al.* 2013).

In recent years, interest in the trend of the elastic modulus value development during the early hardening increased. This is related to the shrinkage strain, especially to the determination of the critical stress inducing the cracks initiation.

Such analysis focused on the determination of the early elastic modulus value was also performed by authors of this chapter. The description of the experiment, results and main conclusions are given in the next paragraphs.

8.5. Materials and methods

Most of the above-described testing methods are designed to determine the modulus of elasticity by making measurements on hardened cement materials. However, in many cases (a need to remove formwork early, installing tensioning cables at ages of less than 4 hours) it is necessary to know the development of the modulus of elasticity of a concrete during the setting and early hardening. The main goal of the experiment described herein was thus to explore the possibilities of determining the modulus of elasticity of cement-based materials during the first 3 days of age. The experiment has several stages, but only the first stage (when cement pastes were tested) is described here. The subsequent stages used cement mortars and later also concretes. However, first, it is good to verify the possibilities of determining the modulus of elasticity using basic materials – in this case cement pastes.

Six cement pastes were made using cement CEM I 52.5 R (Mokrá, Czech Republic), a half of which contained plasticiser Sika ViscoCrete 4035 at an amount of 1 % of cement mass. Pastes without plasticiser had the w/c ratio equal to 0.50, 0.40 and 0.33. The lowest w/c ratio corresponds to the lowest theoretical amount of water required for the cement to fully hydrate. These pastes were identified as 050, 040, and 033. Pastes with plasticiser had the same w/c ratio as the pastes without plasticiser and were named P050, P040, and P033. Each cement paste was made into nine prism-shaped specimens with the nominal dimensions of 40 × 40 × 160 mm and one specimen in the shape of Vicat's ring, see Fig. 8.6. The ring specimen was used for continuous measurement of the dynamic modulus of elasticity during the first 24 hours using an ultrasonic device. The apparatus consists of measurement cell/chamber equipped with the ultrasonic probes and datataker, where the data are stored in the interval of 60 sec. The strength of the signal was set to 2000 V for the purpose of early measurement. The prisms were used for measuring the dynamic and static modulus of elasticity at an age of 24 through 72 hours. Over the first 24 hours the prisms were left in plastic moulds under standard laboratory conditions at an ambient temperature of (22 ± 2) °C and relative humidity of (55 ± 5) %, covered with a PE sheet. At the age of 24 hours they were demoulded, four were non-destructively and later destructively tested and the remaining four were wrapped in the PE sheet and left to age in the same conditions. These other four specimens were used for determining the modulus of elasticity at the age of 48 and 72 hours.

Fig. 8.6. Specimens – 9 prisms of 40 × 40 × 160 mm and 1 Vicat's ring

Once the cement paste was mixed, the next step was filling the Vicat's ring and placing it in the measurement chamber of the ultrasonic measurement device. The apparatus is pictured in Fig. 8.7, refer to (http://www.schleibinger.com) for more details. The ring with the setting cement paste is positioned between two transducers running at 54 kHz, one of which is a transmitter and the other a receiver of the ultrasonic pulse. The instrument continuously records the transit time of ultrasonic waves travelling through the material, which is primarily designed to measure the setting time. Given the fact that the instrument's design ensures a constant distance from the measurement base (40 mm), it is possible to calculate the ultrasonic pulse transit velocity and subsequently also values of the dynamic modulus of elasticity over the observed time period using the following formula (included in the manual to the instrument):

$$E = \rho \cdot v^2 \tag{8.2}$$

Where:

E – dynamic modulus of elasticity in N/mm^2,

ρ – bulk density in kg/m^3,

v – ultrasonic pulse velocity in km/s.

A slight disadvantage of this measurement and subsequent calculation of the dynamic modulus of elasticity is the fact that the equation (eq. 8.2) does not take into account Poisson's ratio, which typically enters the calculation when using the ultrasonic pulse velocity test.

Fig. 8.7. Measurement using the ultrasonic device during setting and early hardening

During the first 24 hours of ageing the modulus of elasticity was thus observed using the ultrasonic apparatus. At ages 24 through 72 hours, other test methods were applied using the prisms as specimens for measurement. As mentioned earlier, the first four specimens were tested for the modulus of elasticity at an age of 24 hours. The dynamic modulus of elasticity was measured again using the ultrasonic pulse velocity test, however, using an ultrasonic device (https://www.proceq.com) as well as using the resonance method. The natural frequencies of longitudinal, flexural, and torsional vibration of the specimens were measured using an oscilloscope with an acoustic emission sensor. The vibration was produced by a mechanical impulse delivered by an impact hammer. Fig. 8.8 shows the measurement of the dynamic moduli of elasticity using the prisms.

The dynamic modulus of elasticity determined by the ultrasonic pulse velocity test was calculated using the following equation:

$$E_{cu} = \rho \cdot v_L^2 \cdot \frac{(1+\mu)\cdot(1-2\cdot\mu)}{1-\mu} \tag{8.3}$$

Where:

E_{cu} – dynamic modulus of elasticity in N/mm^2,
ρ – bulk density in kg/m^3,
v_L – ultrasonic pulse velocity in km/s,
μ – dynamic Poisson's ratio [-].

The dynamic modulus of elasticity was calculated from the natural frequency of longitudinal vibration using the equation:

$$E_{crL} = 4 \cdot L^2 \cdot f_L^2 \cdot \rho \tag{8.4}$$

Where:

E_{crL} – dynamic modulus of elasticity in N/mm^2,
L – specimen length in m,
f_L – natural frequency of longitudinal vibration in kHz,
ρ – bulk density in kg/m^3.

The dynamic modulus of elasticity was calculated from the natural frequency of flexural vibration using the equation:

$$E_{crf} = 0{,}0789 \cdot c_1 \cdot L^4 \cdot f_f^2 \cdot \rho \cdot \frac{1}{i^2} \tag{8.5}$$

Where:

E_{crf} – dynamic modulus of elasticity in N/mm^2,
c_1 – correction coefficient [-],
L – specimen length in m,
f_f – natural frequency of flexural vibration in kHz,
ρ – bulk density in kg/m^3,
i – cross-sectional radius of gyration of a specimen in m.

An advantage of the resonance method is the fact that it can be used to determine the dynamic Poisson's ratio, which enters the calculation of the dynamic modulus of elasticity E_{cu} using equation (eq. 8.3). A slight disadvantage is that the resonance method can only be applied when testing solid materials, cement materials can thus only be measured once they have hardened.

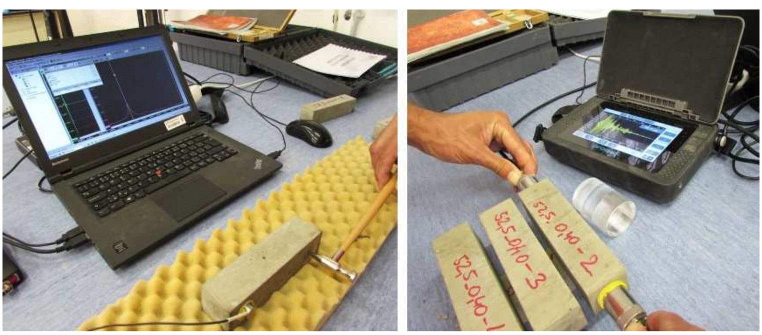

Fig. 8.8. Measurement using the resonance method (left) and the ultrasonic pulse velocity test (right)

After non-destructive measurements the prisms were tested for the static modulus of elasticity at the age of 24 hours. The static test was performed according to standard (ISO 1920-10, 2010) by applying a compressive load onto a specimen while measuring longitudinal strain. The test itself is designed as cyclic, alternating between two loading levels – the basic load is 0.5 N/mm^2 and the top load is equal to 1/3 of the expected compressive strength. For this reason, one specimen was always used to determine compressive strength and the remaining three were then tested for the modulus of elasticity E_c. The specimens were loaded using a hydraulic testing press. Their longitudinal strain was measured along an 80-mm base using strain transducers connected to a data logger (Fig. 8.9). The E_c test was completed by measuring the compressive strength of the specimens.

Fig. 8.9. Measurement of the static modulus of elasticity (left) and a detailed view of the strain transducers (right)

At the age of 48 hours the cement pastes were tested only for the dynamic modulus of elasticity following the above-described methods using the remaining four specimens (the ninth prism was used for the measurement of internal temperature during ageing). Immediately after the end of measurement at the age of 48 hours, the specimens were again wrapped in a PE sheet and left until the age of 72 hours, when they were once again tested for both the dynamic and static modulus of elasticity.

8.6. Results and discussion

The results of measurements of the dynamic modulus of elasticity over the first 24 hours of age using the ultrasonic device with a measurement chamber are shown in Fig. 8.10. The average values of the dynamic moduli of elasticity E_{cu}, E_{crL}, and E_{crf} as well as the static modulus of elasticity E_c are plotted as bar charts in Fig. 8.11 through 8.14, with the error bars representing a sample standard deviation.

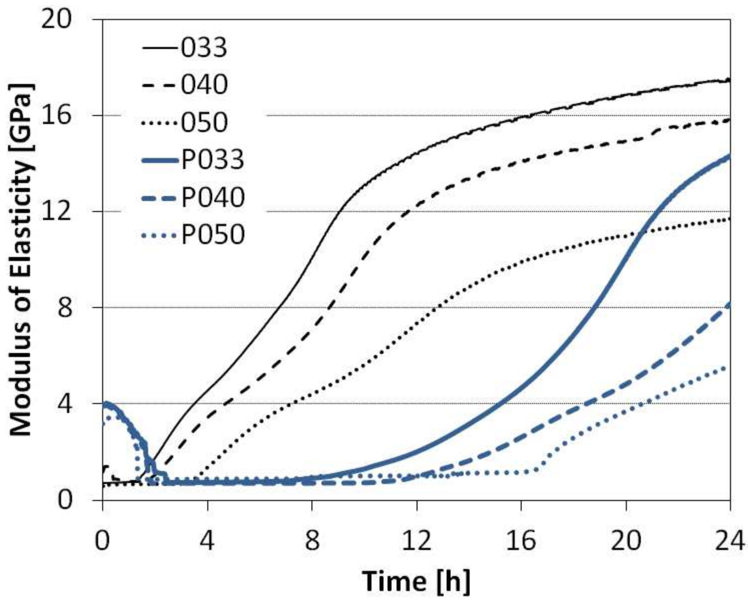

Fig. 8.10. Development of the dynamic modulus of elasticity determined over the first 24 hours, measured using the ultrasonic device with a measurement chamber

The continuous measurement (see Fig. 8.10) captured a very early stage of hydration period (end of the first hydration peak) when the viscosity of the material was changing from the fluid to thixotropic state. This process was very well visible in the case of cement pastes with the plasticizer, when the initial value (at the start of measurement) of the modulus of elasticity corresponded to the ultrasonic pulse velocity of approx. 1420 m/s which was close to the value of the ultrasonic pulse velocity of water. The subsequent changes in viscosity and formation of material's microstructure caused damping of the ultrasonic waves which was reflected in the decrease in the elastic modulus value. During the dormant period there were no changes in the value of elastic modulus. The subsequent growth of the elastic modulus started with the growth of the internal temperature measured inside the test specimens.

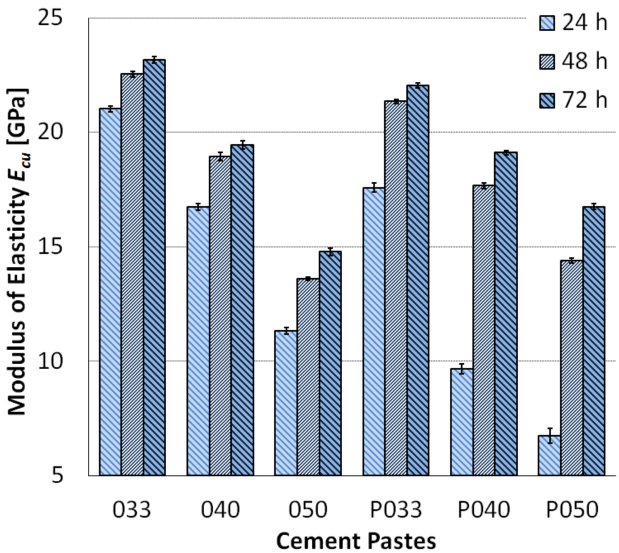

Fig. 8.11. Results of the dynamic modulus of elasticity Ecu at the age of 24, 48, and 72 hours

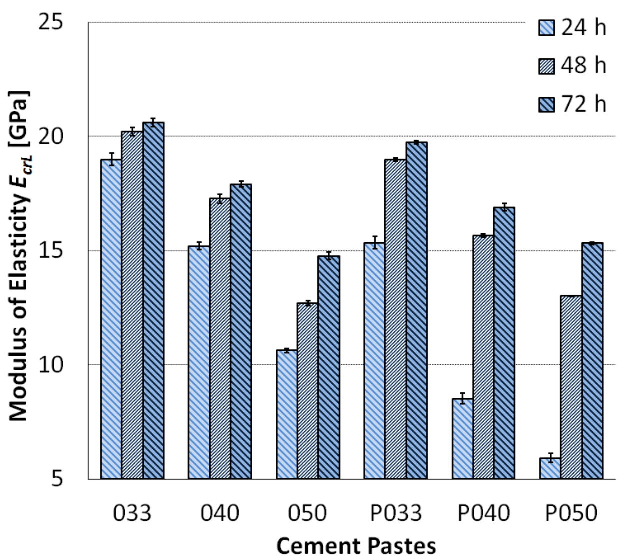

Fig. 8.12. Results of the dynamic modulus of elasticity EcrL at the age of24, 48, and 72 hours

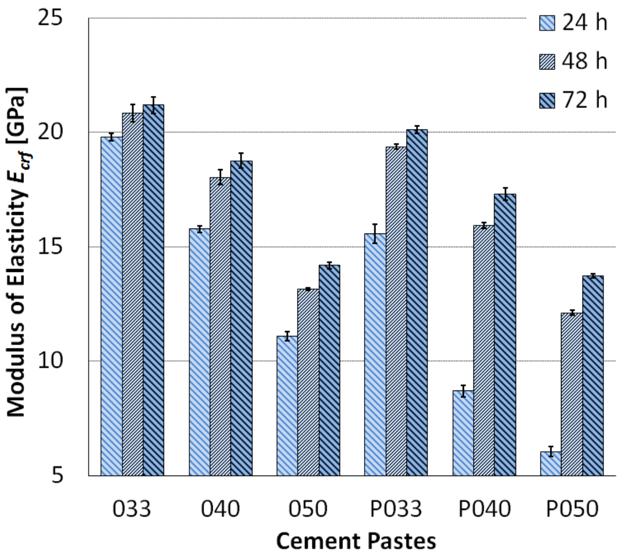

Fig. 8.13. Results of the dynamic modulus of elasticity E_{crf} at the age of 24, 48, and 72 hours

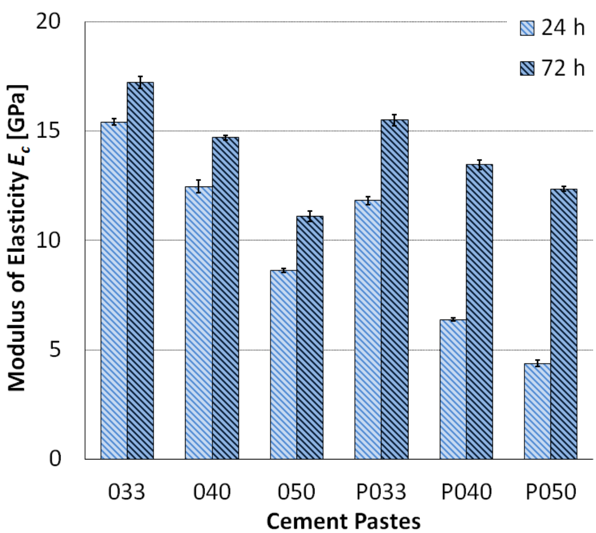

Fig. 8.14. Results of the static modulus of elasticity E_c at the age of 24 and 72 hours

The diagrams above show that the modulus of elasticity in pastes with plasticiser began to increase later than in pastes without plasticiser. This is most clearly visible in the first 24 hours of ageing. The results of the elastic modulus measurements also showed an influence of the w/c ratio, specifically, where the w/c ratio decreased, the modulus of elasticity increased. Aside from technological factors, the experiment also confirmed the influence of testing factors. It was demonstrated that the static modulus of elasticity reaches lower values than the dynamic modulus. In pastes with no plasticiser the ratio between the static and dynamic modulus, measured by the ultrasonic pulse velocity test, was within 0.73 and 0.76; in pastes with plasticiser it was 0.65 to 0.74. The same ratio, only in the dynamic modulus of elasticity measured by the resonance method, was 0.75 to 0.83 in pastes without plasticiser and 0.72 to 0.90 in pastes with plasticiser. Differences between the measured values of the elastic modulus were registered also in the dynamic values – the ratio between the modulus of elasticity determined by the ultrasonic pulse velocity test and the resonance method was approx. 1.05 – 1.10 in pastes without plasticiser and approx. 1.10 – 1.20 in pastes with plasticiser.

The ultrasonic pulse velocity test appears to be a suitable method for determining the development of the elastic modulus, however, it may have problems giving the precise value of the dynamic modulus of elasticity at a certain time. Measurements made by the ultrasonic pulse velocity test are influenced by many factors, e.g., the frequency of the transducers used, measuring base length (i.e. the distance between the transducers), measuring instrument, the material's moisture content, etc., which makes it difficult to obtain an "accurate" value. The results of the experiment showed this fact in the comparison of the values measured by different types of ultrasonic devices (Fig. 8.7and Fig. 8.8) at an age of 24 hours – the results differed by up to 20 %. Moreover, if the calculation of the dynamic modulus of elasticity determined by the apparatus (see Fig. 8.7) included Poisson's ratio, the difference may have been even greater. In fact, the standard (ASTM C597-16, 2016) does not even recommend the calculation of the elastic modulus using data obtained by the ultrasonic pulse velocity test.

8.7 Conclusions

The experiment results showed the elastic modulus of cementitious materials can be determined not only in the hardened state, but also during initial stages when the internal microstructure is still beginning to form. They also confirmed the fact that technological and testing factors have an influence on the value of the modulus of elasticity in cement pastes. Both the w/c ratio and plasticiser content have been proved to have an effect on the development of the elastic modulus in young cement pastes. Pastes, which contained plasticisers, showed significant delays in the development of the modulus of elasticity, especially during the first 24 hours. The fact that the choice of testing method affects the value was also confirmed. Tests made with the prism specimens have shown that the highest values were reached by dynamic moduli of elasticity measured by the ultrasonic pulse velocity test, whereas the lowest values were, as expected, the static moduli. The influence of the specimens (and probably also the testing apparatus) was apparent in the values of the dynamic modulus of elasticity at the age of 24 hours measured by the ultrasonic instrument using Vicat's rings and the another one using prism specimens – the modulus of elasticity measured for the rings reached, depending on the type of paste, approx. 80 to 95 % of the value of the elastic modulus measured on prisms.

In conclusion, the ultrasonic apparatus with a measurement chamber appears to be a suitable tool for determining the dynamic modulus of elasticity during the setting of cement composites. The question remains, however, how to ascertain Poisson's ratio in a setting material, as it also affects the value of the dynamic modulus of elasticity, see equation (eq. 8.3). This would probably require an instrument capable of measuring compressive and shear wave velocity, see e.g. (Carette *et al.*, 2012).

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