Characterizing Materials in the Frequency Domain: Validation Experiments

Josef Katzmann (né Sauer)\textsuperscript{1,∗}, Jana Musialak\textsuperscript{1}, and Holger Steeb\textsuperscript{1}

\textsuperscript{1} Institute of Applied Mechanics (CE), University of Stuttgart, 70659 Stuttgart, Germany

High Performance Concrete (HPC) and Ultra High Performance Concrete (UHPC) achieve an effective stiffness or strength which allows constructing highly stressed buildings. Following, we present a mechanical characterization technique for HPC and UHPC, using harmonic excitations from 0.1 Hz up to 1 kHz and displacement amplitudes in the micrometer range. The small-amplitude excitations are performed by a high-performance piezo-electric actuator. To validate the experimental workflow, we characterize the mechanical behaviour of Polymethylmethacrylat (PMMA). In this contribution, we present results gained with two different cylindrical sample sizes. By this comparison we shall ensure that geometrical scaling of samples has no influence on the rheological results of the experiments, apart from a change in Signal-to-Noise ratio.

1 Motivation

The fatigue strength of concrete, i.e. cycle-dependent secant moduli, can be determined by well-established low-cycle fatigue experiments. Such experiments are described for example in [2]. As also shown [2], for this low-cycle fatigue no frequencies above 200 Hz are considered usually. Our aim is to increase the excitation frequency to the kHz regime, which allows us to a) characterize the cycle-dependent moduli in significantly smaller periods and b) to obtain material information at higher frequencies.

2 Material and Method

Fig. 1 displays a sketch of the setup for the oscillation measurements. Likewise setups have been applied in the past for various geophysical studies, as recently reviewed [5]. In our case, the axial stress is measured by the aluminium (Al) standard placed directly below the sample. Fig. 2 shows an example of the raw axial stress and axial strain signals obtained at 10 Hz. All signals are recorded and processed by the measuring amplifier (HBM QuantumX MX410B).

The setup is mounted into a stiff universal testing machine (Schenk RM50, extended by a DOLI EDC controlling unit). This apparatus creates a static preload, around which oscillates the piezo-electric actuator (Physik Instrumente P-235.20).

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig1.png}
\caption{Sketch of the experimental setup.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{fig2.png}
\caption{Example of axial stress and axial strain signals in time domain.}
\end{figure}

∗ Corresponding author: e-mail josef.katzmann@mechbau.uni-stuttgart.de
The waveform generator (Agilent 33500B) produces the harmonic signal, which is amplified by the high-voltage amplifier (PI E-482). To obtain the sample’s effective visco-elastic properties, at first we sweep over an amplitude range at different frequencies (1, 10, 100 and 1000 Hz). Then, after choosing a certain region within this amplitude range where the visco-elastic properties show no dependence of amplitude, we perform frequency sweeps.

As material, we use two cylindrical PMMA samples. Typical properties for this material are a static Young’s modulus of 3.3 GPa and a static Poisson’s ratio of 0.37 [www.plexiglas.de]. Both samples have an aspect ratio, i.e. length per diameter, of 2.5. The smaller sample ("small" or "s") has 12 mm in diameter, the bigger sample 30 mm ("normal" or "n", since this is a typical value for studies already documented [3].

3 Results and Discussion

3.1 Amplitude Sweeps

Fig. 3 and Fig. 4 display the amplitude-dependent Young’s modulus magnitude and the amplitude-dependent Poisson’s ratio magnitude, respectively. The former one remains nearly constant for sample ‘n’, for sample "s" it does so above medium high amplitudes. Its values increase with frequency, while they are roughly similar per frequency for both samples. The absolute values of Young’s modulus magnitude lie between 3.7 and 5.7 GPa, with a variation of about 2 % between the samples. A reason why at smaller amplitudes the values for "s" are fluctuating more than for "n" could be that due to the smaller diameter, the same strain amplitudes cause smaller force amplitudes, so the Signal-to-Noise ratio is smaller.

Poisson’s ratio magnitude stays constant for all amplitudes and for both samples. It decreases with frequency. Between the samples, the values show some deviation: For example, Poisson’s ratio magnitude for "s" at 1 Hz lies close to the curve for "n" at 10 Hz. The absolute values reach from 0.335 to 0.375, with roughly 3 % variation between "s" and "n".

Judging from these amplitude sweeps, strain amplitudes of about $4 \cdot 10^{-5}$ and $2 \cdot 10^{-5}$ are chosen for "s" and "n", respectively, for the following frequency sweeps. These domains of linear amplitude are highlighted in Fig. 3 and Fig. 4.

![Fig. 3](image1.png)  
**Fig. 3:** Amplitude-dependent Young’s modulus magnitude.

![Fig. 4](image2.png)  
**Fig. 4:** Amplitude-dependent Poisson’s ratio magnitude.

3.2 Frequency Sweeps

Fig. 5 to Fig. 8 show the frequency-dependent Young’s modulus and the frequency-dependent Poisson’s ratio, respectively. For comparison, experimental results from [1, 3, 4, 6] (abbreviations "Batz06", "PimForGue15", "PimForGue16" and "YeeTak82", respectively) are plotted. Frequency-dependent Young’s modulus magnitude increases linearly with frequency. The results for "s" and "n" are nearly identical. The slope of increase is about 0.5 GPa/dec. In shape and values, the measured curves mostly show a good agreement with the curves taken from literature. Only "Batz06" differs in absolute values, but agrees in slope.

Frequency-dependent Young’s modulus loss factor increases until 10 Hz and decreases for frequencies above. Again, the curves for "s" and "n" are close together. The absolute values range from 0.03 to 0.075. From the literature results, "PimForGue15" shows the closest match to the data measured in our investigations.

Frequency-dependent Poisson’s ratio magnitude decreases linearly with frequency. The slope of decrease is about 0.01/dec. The absolute values lie between 0.33 and 0.385. The curve for "n" is shifted to higher values by an offset of 0.01 to the curve
for "s". The curve "PimForGue15" shows the same decrease, but is shifted by an offset of 0.01 to the curve for "n". The values from "YeeTak82" lie between the measured results, with a slope of decrease of 0.005/dec.

The measured results of frequency dependent Poisson’s ratio loss factor are inverted for the comparison with literature. They also increase until 8 Hz for "n", or until 10 Hz for "s", i.e., the curve for "n" is shifted to smaller frequencies compared to "s". Above these critical frequencies, both curves decrease. The absolute values range from 0.013 to 0.021. The values from "PimForGue16" lie close to "n". The values from "YeeTak82" are noticeably smaller, but also show a peak at 10 Hz.

### 3.3 Comparison with the Generalized Maxwell Model

In this section, a simple generalized Maxwell model is used to approximate the measured complex Young’s modulus \(E^* = E' + iE''\). This property is now displayed as real part (storage modulus) in Fig. 9 and imaginary part (loss modulus) in Fig. 10.
For the Generalized Maxwell model, the real part and the imaginary part of Young’s modulus can be calculated as

\[ E' = E_\infty + \sum_{i=1}^{N} \left( E_i \frac{(f/f_i)^2}{1 + (f/f_i)^2} \right) \quad \text{and} \quad E'' = \sum_{i=1}^{N} \left( E_i \frac{f/f_i}{1 + (f/f_i)^2} \right), \]

respectively. In these equations, \( E_\infty \) stands for the equilibrium Young’s modulus, \( f_i \) denotes the characteristic frequencies, \( E_i \) stands for the associated Young’s moduli and \( N \) is the number of Maxwell chains used. Here, \( E_\infty \) is chosen as 3.7 GPa. The other model parameters are listed in Tab. 1. With these parameters, we achieve a close approximation to the measured values.

| Index \( i \) | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    |
|---------------|------|------|------|------|------|------|------|------|------|
| Characteristic frequency \( f_i \) [Hz] | 0.100 | 0.316 | 1.00 | 3.16 | 10.0 | 31.6 | 100  | 316  | 1000 |
| Young’s modulus \( E_i \) [GPa]    | 0.18  | 0.20  | 0.22  | 0.24  | 0.28  | 0.24  | 0.22  | 0.20  | 0.18  |

4 Conclusion

For validation of our experimental setup and method, we have used two PMMA cylinders, one with dimensions typical for geophysical studies and one significantly smaller. Comparing the visco-elastic Young’s moduli and Poisson’s ratios, we have obtained quite similar (physically plausible) results. Furthermore, we noticed a peak frequency of about 10 Hz for loss factors. Finally, we have fitted the measured values with simple model curves. In future experiments, we can use the presented setup and method to characterize HPC, UHPC and other materials showing small attenuation.

5 Acknowledgements

Financial support was provided by the German Science Foundation (DFG) in the framework of the project “Temperature and humidity induced damage processes in concrete due to pure compressive fatigue loading” of the DFG priority program SPP 2020 through grant number STE969-12/1. This support is gratefully acknowledged.

References

[1] M. L. Batzle, D. H. Han, and R. Hofmann, Geophys. 71(1), N1 (2006).
[2] N. Oneschkow, Analyse des Ermüdungsverhaltens von Beton anhand der Dehnungsentwicklung (German), PhD thesis, (Institut für Baustoffe, Leibniz Universität Hannover, Hannover, 2014).
[3] L. Pimienta, J. Fortin, and Y. Guéguen, Geophys. 80(5), L57 (2015).
[4] L. Pimienta, J. Fortin, and Y. Guéguen, Geophys. 81(2), D183 (2016).
[5] S. Subramaniam, B. Quintal, N. Tisato, E. H. Saenger, and C. Madonna, Geoph. Prosp. 62, 1211 (2014).
[6] A. F. Yee, and M. T. Takemori, J. Polym. Sci., Polym. Phys. Ed. 20, 205 (1982).