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NONDESTRUCTIVE DETERMINATION OF COMPRESSIVE STRENGTH OF CALCIUM ALUMINATE CEMENT MATERIAL

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Abstract: Calcium aluminate cement (CAC) mortar was investigated by nondestructive ultrasonic measurement in the through-transmission mode and compressive strength measurements. The detected profile of ultrasonic signal was fitted as a sum of two functions of the following form: $U(t)=A \sin(\omega t+\phi) \exp(-(t-tc)2/w2)$. From the fitted ultrasonic wave signal one obtains wave packets group velocity, angular frequency and envelope amplitude. The product of those parameters is proportional to the stress induced in the cement material and it was found experimentally to be proportional to the compressive strength of the material.

1. INTRODUCTION

Nondestructive methods of investigation of cement materials are especially attractive nowadays because of the continuous need to evaluate the state of the concrete structures already in use [1]. There is a long record of the use of ultrasonic NDT (Non-Destructive Testing) methods on concrete [2, 3] but the fundamental relationship between the ultrasonic parameters extracted (primarily connected with elastic properties of material) and compressive strength of material is still lacking. In our opinion it is not only the consequence of the complex structure of cement material (composite porous body containing microcracks and pore fluids) and the rich variety of wave - micro-structure interactions (reflection, attenuation, wave conversion) but also because of only partial use of the ultrasonic wave information content. This work aims to contribute in both directions, both by compressive strength measurement of cement material and utilizing the parameters obtained through appropriate description of large portion of the transmitted ultrasonic signal [4]. From the simple model of one dimensional longitudinal ultrasonic wave propagation [5] it is shown that such a combination of ultrasonic parameters is possible, and the extracted property of ultrasonic pulse/disturbance propagation in material have the same units of measurement as the compressive strength.

2. THEORETICAL BACKGROUND

The simple one dimensional model of longitudinal ultrasonic wave propagation [5] connects the relative deformation of the material caused by the displacement of one of its end with particle velocity, vp and wave velocity, c (see Fig. 1):



Figure 1. Longitudinal wave velocity propagation model

Only the portion of the material of the length $c\Delta t$ is under stress, and its relative deformation is:

$$\varepsilon = \frac{\sigma_x}{E} = \frac{\Delta l}{c\Delta t} = \frac{v_p}{c} \tag{1}$$

Since particle velocity is proportional to amplitude and angular frequency of oscillation, and wave velocity is dependent on elastic modulus and material density, after simple rearrangements, it follows that stress of the material is given by Eq. 2:

$$\sigma_x \propto A \omega v_G \tag{2}$$

Basically, we rely on wave equation [3, 5, 7, 8] and not on the more complex/complete the so-called telegraphic equation which includes damping force $(p(\partial u/\partial t))$ and wave dispersion term (qu).

3. METHODS

The CAC was taken from a regular production of "Istra Cement International", Pula, Croatia, part of Heidelberger Zement Group. The cement has the following oxide mass fraction composition: CaO, 40.2%, Al₂O₃, 39.0%, Fe₂O₃, 11.7%, FeO, 4.3% and SiO₂, 1.9%. The principal mineral phase is monocalcium aluminate, CaAl₂O₄, with mayenite, belite and ferrite phase as minor phases. The CAC mortar was prepared at w/c ratio of 0.30 and 20°C in the Laboratory of the Faculty of Chemical Engineering and Technology as described previously [6]. The standard quartz sand to cement ratio was 3:1. Samples were investigated by ultrasonic measurements in through-transmission mode under immersion in the Laboratory for Non-destructive Testing at Faculty of Mechanical Engineering and Naval Architecture - University of Zagreb (Fig. 2) and by compressive strength determination according to HRN EN 196-1. The ultrasonic probes used were K0.5S/B0.5SL operating at 500 kHz frequency and were driven by spike impulse at a repetition frequency of 100 Hz. Transient signals were measured using LeCroy 9310 AM 400 MHz oscilloscope.



Figure 2. Ultrasonic measurement setup

4. **RESULTS AND DISCUSSION**

The early hydration of the prepared CAC mortar was investigated by NDT ultrasonic measurement of longitudinal ultrasonic waves in through-transmission mode and compressive strength measurements. During that time, within 7 h, the physical appearance of the sample changes from easily flowing material to the cement stone with compressive strength of more than 45 MPa (see Table 1).

Time of hydration / h	Compressive strength / MPa
2:26	<0.2
3:03	10.3
3:33	17.8
4:03	25.6
4:33	31.9
5:03	38.8
5:33	39.4
6:03	47.5
6:33	47.8

Table 1. The experimental data of compressive strength measurement.

The evolution of the ultrasonic signal with the advancement of hydration reactions is shown in Fig 3. It is seen that the advancement of hydration is accompanied by increase of the amplitude of the signal as well as the decrease of the time of arrival.



Figure 3. Comparison of the ultrasonic signals at different hydration times (absolute signal values are shown).

The sample of measured ultrasonic signal is shown in Fig 4 at 6:38 hours of hydration. Only the portion of the signal corresponding to the arrival times less than the arrival of the first echo from the sending/generating transducer is taken into account for further data analysis.



Figure 4. Explanation of the portion of ultrasonic signal used for data fitting.

The whole signal thus obtained is fitted as a sum of two wave packets (see Fig. 3) and the results are shown in Table 2.

Table 2. Results of the fitting the ultrasonic signal on the pr	oposed model:
$U(t) = A_1 \sin(\omega_1 t + \varphi_1) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_1^2) + A_2 \sin(\omega_2 t + \varphi_2) \exp(-(t - t_{C1})^2 / w_2) \exp(-(t - t$	$D(-(t-t_{C2})^2/w_2^2)$

<i>t /</i> h	A_1 / V	<i>∞</i> 1 /Mrad/s	<i>t</i> _{C1} / μs	$A_1\omega_1/t_{\rm C1}$	A_2 / V	∞ ₂ /Mrad/s	t _{C2} / μs	$A_2\omega_2/t_{\rm C2}$
02:07	0.01904	2.54656	14.91209	0.00325	0.00229	2.43159	21.58303	2.58E-4
02:11	0.05543	2.56555	14.88238	0.00956	0.01057	2.39359	21.32859	0.00119
02:15	0.10214	2.60332	14.4908	0.01835	0.02164	2.61744	21.50734	0.00263
02:21	0.15819	2.65264	14.18947	0.02957	0.04079	2.8009	21.10205	0.00541
02:28	0.24411	2.7102	13.88569	0.04765	0.06807	2.93889	20.82138	0.00961
02:30	0.26944	2.72035	13.79175	0.05315	0.07586	2.97483	20.67727	0.01091
02:37	0.36796	2.77031	13.4840	0.0756	0.0993	3.02613	20.15979	0.01491
02:40	0.39844	2.78166	13.41536	0.08262	0.11007	3.03337	20.09203	0.01662
02:47	0.52374	2.82065	13.07472	0.11299	0.13289	3.07772	19.4405	0.02104
02:55	1.9964	2.92543	12.68218	0.46051	0.56148	3.0731	18.65193	0.09251
03:02	2.61593	2.96573	12.54615	0.61837	0.7105	3.06734	18.44438	0.11816
03:13	5.8844	2.99304	12.39773	1.4206	0.84725	3.24392	18.25959	0.15052
03:25	6.91378	3.02713	12.29711	1.70194	0.98932	3.2392	18.00332	0.17800
03:27	7.03211	3.00521	12.21496	1.73009	0.84906	3.16643	19.55937	0.13745
03:37	7.45299	3.00566	11.89993	1.88246	1.12816	3.17324	18.94381	0.18898
03:40	7.77508	3.00464	11.85727	1.97021	1.06821	3.1617	18.66313	0.18096
03:50	8.56676	3.04038	11.78816	2.20952	1.13911	3.26063	17.98901	0.20647
03:59	9.53587	3.07359	11.55819	2.53581	1.28747	3.20949	18.76468	0.22021
04:07	10.24609	3.09497	11.34842	2.79434	0.83745	2.40608	18.78977	0.10724
04:17	11.40805	3.07083	11.33211	3.09141	0.90107	2.59638	18.50624	0.12642
04:25	12.11577	3.10063	11.2484	3.33972	0.65625	2.58983	17.90977	0.0949
04:32	12.70636	3.08664	11.1648	3.51282	0.44333	2.6619	17.58252	0.06712

04:46	13.69684	3.10478	11.10106	3.83078	0.52789	3.49198	15.4169	0.11957
05:07	15.4432	3.0842	11.07712	4.29985	1.66428	2.9578	14.99357	0.32831
05:15	16.1601	3.12357	10.93898	4.61443	1.84627	3.23534	14.47713	0.4126
05:24	16.7782	3.11865	10.77116	4.85791	2.70856	3.09026	14.84483	0.56384
05:32	17.50655	3.13761	10.72854	5.11987	2.15947	3.05471	14.7725	0.44654
05:53	17.75809	3.11395	10.73304	5.15211	2.45424	3.08458	14.78605	0.51199
06:13	18.06183	3.14321	10.65596	5.32773	2.76292	3.1207	14.6041	0.5904
06:38	18.31474	3.13196	10.53842	5.44304	3.07396	3.12719	14.36058	0.66939
06:47	19.3339	3.12623	10.53115	5.73937	3.53471	3.09877	14.31359	0.76523
07:10	19.5613	3.1392	10.58049	5.80378	3.31513	3.11541	14.32619	0.72092
07:18	19.52169	3.12468	10.45582	5.83398	3.67491	3.14867	14.1119	0.81995
07:27	19.99414	3.13538	10.43888	6.00536	3.67224	3.14533	14.06574	0.82117
07:35	20.38005	3.13397	10.45235	6.11063	3.99252	3.11024	14.08228	0.8818

Coefficient of correlation, R^2 is typically greater than 0.997, and example of the fit for the ultrasonic signal at 7:35 h is shown in Fig 5. Absolute values of the signal are shown for better clarity/visibility. Standard deviation is less than 1% of the signal span. From the Fig 5 it is seen that the first wave packet is faster and has greater intensity, so in our further work we have concentrated on the parameters of the first wave packet. Simple one-dimensional model for the propagation of longitudinal ultrasonic waves [5] have shown that the product of the group velocity, angular frequency and signal envelope is proportional to the magnitude of the stress accompanying the propagation of the disturbance/wave packet. It is shown experimentally that the compressive strength of the cement material studied is proportional to the $A\omega v_G$, as shown on Fig 6.



Figure 5. Example of the fit for the ultrasonic signal at 7:35 h is shown. Absolute values of the signal are shown for better clarity/visibility.



Figure 6. Development of compressive strength (as determined in standard compressive test) and of the product of group velocity, angular frequency and signal envelope. Results of measurement of compressive strength and the product $A\omega v_{G}$.

5. CONCLUSION

The CAC mortar samples investigated gain compressive strength greater than 45 MPa within 7 hours and could be used for preparation of rapid setting and hardening cement material suitable for emergency repairs. The advancement of the hydration process is monitored indirectly, through the compressive strength measurements and nondestructive ultrasonic through-transmission measurements. The large portion of ultrasonic signal could be successfully fitted as a sum of two functions of the following form: $U(t)=A \sin(\omega t+\varphi) \exp(-(t-t_c)^2/w^2)$ and extract the properties of the two wave packets, especially group velocity, angular frequency and envelope amplitude. The application of simple one-dimensional model for the propagation of longitudinal ultrasonic waves has shown that the product of the aforementioned quantities is proportional to the magnitude of the stress accompanying the propagation of the disturbance/wave packet. It is shown experimentally that the compressive strength of the cement material studied is proportional to the $A\omega v_G$, i.e.

$$\sigma/MPa \propto A\omega v_G \propto A_1\omega_1/t_{C1}$$

Since the first wave packet has greater amplitude (and is faster) it is advantageous to use it in practice. This wave we connect with the passage of the longitudinal wave. It is worth noting that in the correlation proposed both physical quantities that are correlated have the same measurement units.

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7. **REFERENCES:**

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