Mean Grain Size Determination in Marbles by Ultrasonic Techniques

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Abstract. In this work, we have studied mean grain size determination of some marbles by using ultrasonic velocity measurements and the ultrasonic relative attenuation (URA) method. We have determined the mean grain size of marbles by using velocity-grain size and echo height-grain size master graphs. By using these values, we have compared the mean grain size of marble samples which were obtained by optic microscope images. The results show less than 10% difference compared with values obtained by an optic microscope. Also, comparison of mean grain size values of marbles determined by ultrasonic methods is given.

1. Introduction

Ultrasonic grain size determination of solid materials can be performed by several techniques which are dependent on ultrasonic quantities such as ultrasonic attenuation, ultrasonic backscattering, and velocity. Ultrasonic materials characterization of solid media by using measurement of either wave speed or attenuation was first reported by Mason and McSkimin [1] in the late 1940's which were using attenuation and scattering of high frequency sound waves.

Properties which have caused attenuation of ultrasound waves in solid samples also have been accepted as mechanical properties of industrial materials. For example, in polycrystalline materials grain size has affected ultrasonic attenuation, velocity, material strength and so on. Ultrasonic grain size determinations have been carried out by several techniques which are dependent on ultrasonic quantities.

The ultrasonic attenuation method has been described in several papers [2-7]. By this method, the amplitudes of successive backwall echoes are used to determine the attenuation coefficient of samples. According to Roney's model, relation between grain size and attenuation is given as

$$\frac{\bar{D}_2}{\bar{D}_1} = \frac{f_1}{f_2} = \frac{\alpha_1}{\alpha_2} \tag{1}$$

where α is the ultrasonic attenuation, f is the frequency and $\bar{D}$ is the mean grain size. According to Eq.(1), $D-\alpha$ graphs of reference samples will provide determination of mean grain size of the other samples. In this technique, we have to be careful in determination of attenuation of all samples and determination of mean grain size of reference samples. Also, the reference samples are needed in ultrasonic velocity methods. Ultrasonic velocity depends on structure and especially is affected by scattering of ultrasonic waves by structure. This ultrasonic velocity technique depends on the velocity–to-grain size relation, which is given by Hirsekorn [8], and he has also described the scattering factor, longitudinal and transverse ultrasonic waves by means of grain size and wave number. Experimental data are given in Refs. [9,10].

One of the these new methods –called URA (Ultrasonic Relative Attenuation)[11] - depends on the first backwall echoes of different samples. The determination of the mean grain size according to reference samples is possible and the main factors that are necessary for the reliability of methods are

(a) The input energy of ultrasonic pulse should be constant for all samples.
(b) The contribution of absorption in total attenuation has to be negligible.
(c) Thickness of all samples should be the same.

One additional method which uses ultrasonic backscattering in determination of grain size was studied by several researchers either theoretically or experimentally [12-15]. In this method, grain noise signals which in the time domain are to register between the backwall echoes are used for the grain size determination. Ultrasonic backscattering method requires more detailed experimental set-up, which requires signal rectification, digitization, averaging, etc. Also this method is fundamentally used for relatively fine grain materials.

2. Samples

Marble samples have been prepared at two marble companies, Bicici Marble Company and Gurel Marble Company (Afyon, Turkey), which are usable for tiles and home decoration. Thickness of the samples was approximately 1 cm and their front surface is a $5\times5\ \text{cm}^2$.

Marble types have been chosen according to differences in porosity and density. Marble types and some physical and chemical properties are listed in Table 1. There are two samples, which have been obtained from different characteristics. The accuracy of the thickness measurement of the samples is 0.05 mm.

Table 1. Some physical and chemical properties of marble samples [16].

Marble Type	Hardness (Mohs)	Density (gr/cm^3)	Specific Weight (gr/cm^3)	Elasticity Modulus (10^4 kg/cm^2)	Porosity (%)	MgO (%)	CaO (%)	Fe_2O_3 (%)	SiO_2 (%)
Eskisehir Beige	3-5	2.71	2.74	1.90	0.49	1.39	52.09	0.33	1.89
Aksehir Black	4-5	2.66	2.69	6.09	0.40	0.49	55.40	0.30	Few
Rosso Levanto	3	2.61	2.72	5.38	3.20	26.25	13.75	9.70	28.35
Rosalia Pink	4	2.70	2.72	2.09	2.90	2.20	52.60	0.45	1.20
Suprene Salome	3-4	2.74	2.76	4.94	0.30	1.42	53.25	0.57	0.56
Verde Laguna	3-5	2.73	2.75	5.19	0.28	1.74	48.63	0.64	4.97
Ameretto White	4	2.70	2.73	6.91	5.60	4.17	50.31	0.32	0.14

3. Experiments

Ultrasonic longitudinal velocity experiments have been conducted at TUBITAK, Marmara Research Center. We have used Krautkramer USN 52L type ultrasonic flaw detector and 5 MHz T/R probe which was product of Automation Industries Inc. We have obtained velocity value directly by using properties of flaw detector. Initially, we have measured thickness of samples with 0.05 mm sensitivity. When we have equalized the interval of front surface and first back-wall echoes to the thickness of samples, the flaw detector shows velocity value on the screen. We have realized measurements eight times for each sample and at the end we have taken mean value for each sample. Our flaw detector is sensitive to 0.1 mm thickness, so we could not determine three samples correctly because of their thickness. We could determine them by 5% error. Velocity measurements and mean values are listed in Table 2.

For URA method, we have used a C-scan system which is the Ultrapac II model of Physical Acoustic Corporation at TUBITAK Marmara Research Center. The transducer was operating at 5 MHz in immersion technique. Along the experiments, we provided a constant water-path and constant input energy. The height of first backwall echo was shown in Table 3. In these measurements we have used the rectified RF (VIDEO) signal display of the system and we get wave forms as shown in Fig 1.

Table 2. Velocity measurements and mean values of marble samples.

Marble Type	Mea. 1 (m/s)	Mea. 2 (m/s)	Mea. 3 (m/s)	Mea. 4 (m/s)	Mea. 5 (m/s)	Mea. 6 (m/s)	Mea. 7 (m/s)	Mea. 8 (m/s)	Mean Velocity (m/s)
Eskisehir Beige	4695	4674	4795	4756	4716	4733	4681	4630	4710
Aksehir Black G	4994	4956	5007	5079	4954	4919	5026	4921	4982
Aksehir Black B	5036	5094	4952	4936	4989	5024	4949	5101	5010
Rosso Levanto	5252	5236	5276	5194	5212	5241	5261	5224	5237
Rosalia Pink	5294	5356	5307	5279	5354	5319	5326	5349	5323
Suprene Salome	5901	5925	5885	5873	5938	5912	5869	5857	5895
Verde Laguna	5857	5849	5919	5923	5934	5929	5885	5904	5900
Ameretto White	6274	6217	6299	6284	6185	6226	6274	6225	6248

Table 3. First backwall echo height of marble samples.

Marble Type	Echo height (% screen height)
Eskisehir Beige	49.0
Aksehir Black G	39.5
Aksehir Black B	37.5
Rosso Levanto	34.5
Rosalia Pink	32.0
Suprene Salome	15.5
Verde Laguna	12.5
Ameretto White	8.0

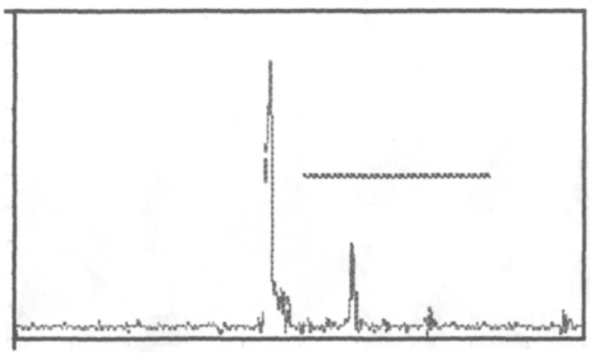


Fig.1. Rectified RF wave form for URA method.

Photomicrographs of marble samples are shown in Fig. 2.a-h. These photomicrographs are taken by Olympus PMG3 model optic microscope, which is connected to Leco 2001 image analyzer. Optic microscope is maintained in Institute of Metallurgy in Osmangazi University. In each sample, image analyzer has detected mainly two different phases.

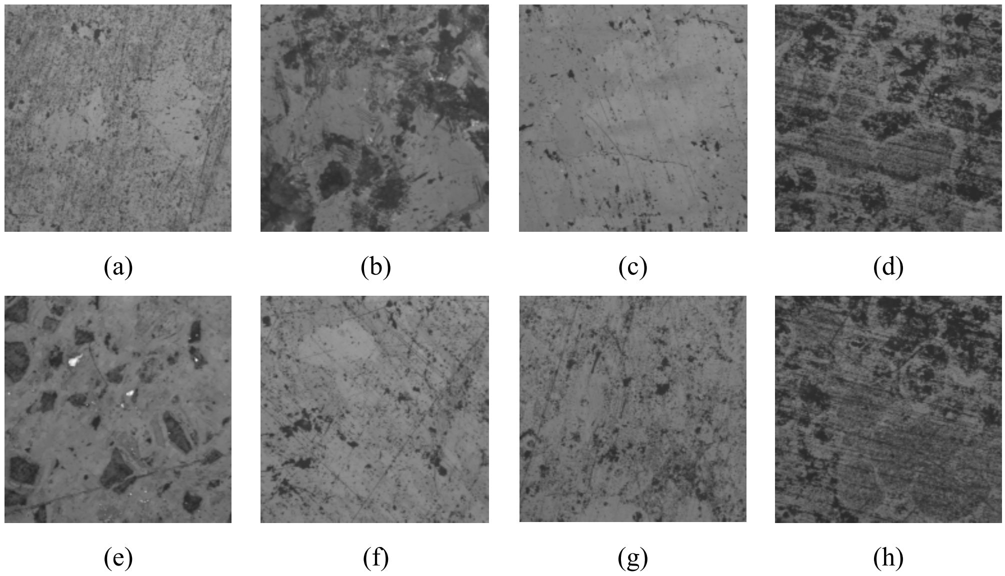


Fig. 2. Photomicrographs of marble samples (a) Eskisehir Beige, (b)Aksehir Black G, (c) Aksehir Black B, (d) Rosalia Pink, (e) Rosso Levanto, (f) Suprene Salome, (g) Verde Laguna, (h) Amaretto White.

4. Results

Mean grain size of samples which are determined by optic microscope are listed in Table 4. Mean grain size values are rounded off to one decimal.

Table 4. Mean grain size of marble samples by optic microscope.

Marble Type	Mean grain size (µm)
Eskisehir Beige	43.9
Aksehir Black G	54.3
Aksehir Black B	55.7
Rosso Levanto	65.3
Rosalia Pink	68.4
Suprene Salome	89.8
Verde Laguna	91.1
Ameretto White	103.1

By using Tables 3, 4 and 5, we have plotted velocity-mean grain size and first backwall echo height-mean grain size graphs which are given in Fig 3.a,b, respectively.

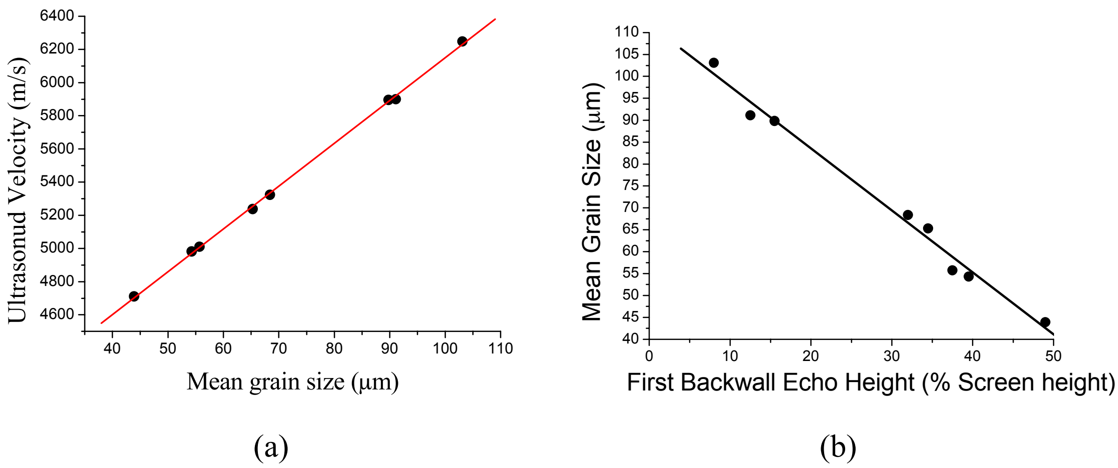


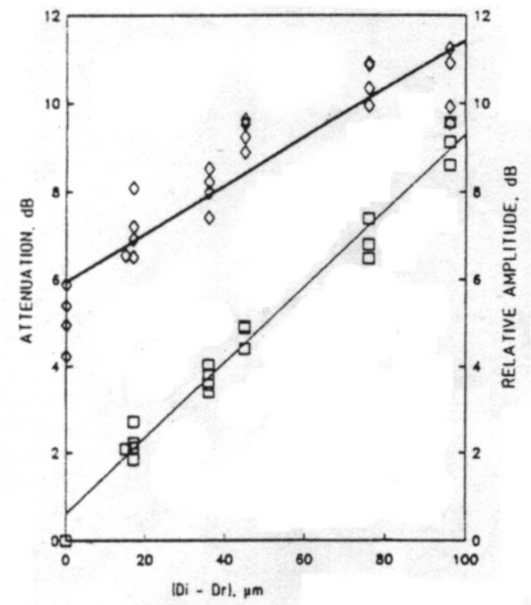
Fig. 3. Master graphs for (a) ultrasonic velocity and (b) URA methods.

According to the [11], the master graph is given as in the Fig.4.a by employing the URA methods. Also we have plotted same graph for our samples.

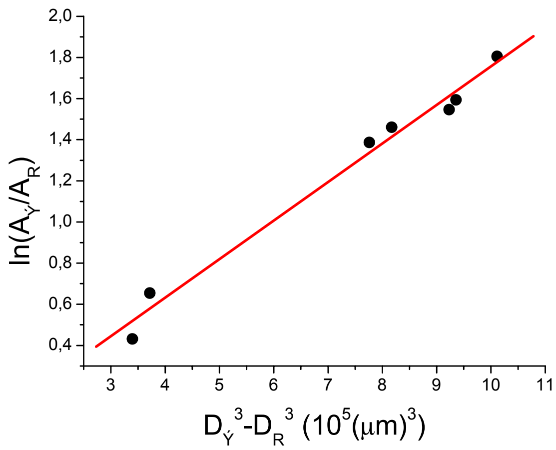
These two graphs show some similarity, but we couldn't see relation between mean grain size and first backwall echo height. Therefore we have plotted Fig. 3.b which indicates this relation.

For ultrasonic velocity method, from Fig. 3.a, correlation coefficent is found to be 0.99966 where the equation of the correlation line is found to be $y = 25.814x + 3570.7$. By using this equation and different velocity values of obtained from other samples of same marble types, we calculated mean grain size of samples, which are given in Table 5.

For URA method, from Fig. 3.b, correlation coefficient of 0.99372 is found and the straight line describing the linear relationship of the correlation is found to be $y = 111.86185 - 1.41486x$. By using this equation and different first backwall echo height values of obtained from other samples of same marble types, we have determined mean grain size values of the samples which are given in Table 5.



(a)



(b)

Fig. 4. (a) URA method's (□) graph of stainless steel samples [11], (b) Our samples graph.

Table 5. Calculated mean grain size of marble samples by ultrasonic methods.

Marble Type	Experimental Mean Grain Size (μm)	Ultrasonic Velocity Method			URA Method		
		Ultrasound Velocity	Calculated Mean Grain Size (μm)	Difference (%)	First Backwall Echo Height (% Screen height)	Calculated Mean Grain Size (μm)	Difference (%)
Eskisehir Beige	43.9	4669	42.5	3.2	48.5	43.2	1.6
Aksehir Black G	54.3	4997	55.3	1.8	39.5	56.0	3.1
Aksehir Black B	55.7	4974	54.4	2.3	37.5	58.8	5.6
Rosso Levanto	65.3	5196	63.0	3.5	34.5	63.0	3.5
Rosalia Pink	68.4	5348	68.9	0.7	33.0	65.2	4.7
Suprene Salome	89.8	5816	87.0	3.1	14.5	91.3	1.7
Verde Laguna	91.1	5983	93.4	2.5	13.0	93.5	2.6
Ameretto White	103.1	6165	100.5	2.5	7.5	101.3	1.7

As shown in Table 5, the differences between mean grain size of experimental and calculated values are less then 4% for ultrasonic velocity method and less than 6% for URA method. As we see from Table 5, URA method shows less accuracy than ultrasonic velocity method. In [11], one of the conditions for the applicability of method is the thickness of samples to be the same. However the thickness of samples in our study is not the same as shown in Table 6, the sample thickness and the difference of the mean grain size value from the experimental values are listed. When the difference is large, the thickness of the sample is far from 10 mm, could be noticed easily.

Calculated mean grain size of samples graph versus experimental value for both methods is given in Fig. 5.

Table 6. Sample thickness and difference between experimental and calculated mean grain size of marble samples in URA method.

Marble Type	Thickness (mm)	Difference (%)
Eskisehir Beige	10.10	0.7
Aksehir Black G	10.00	1.7
Aksehir Black B	9.40	3.1
Rosso Levanto	9.95	2.3
Rosalia Pink	9.85	3.2
Suprene Salome	10.00	1.5
Verde Laguna	10.05	2.4
Ameretto White	10.00	1.8

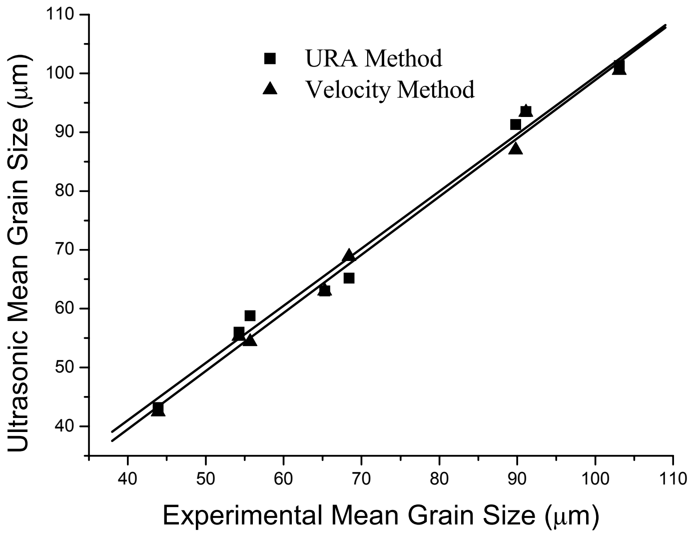


Fig. 5. Experimental vs. Calculated mean grain size graph of marble samples.

5. Conclusion

This work has shown that ultrasonic velocity measurements can be used to estimate mean grain size in marble. In the presented work another non-destructive ultrasonic method has been used to determine the mean grain size of marbles.

It can be seen in Fig. 5, that both of the two ultrasonic methods give closer results to each other when the grain size of samples are absolutely larger. However, when the grain size of the samples becomes smaller, a difference between the values obtained by two methods is occurred. The ultrasonic velocity methods have given more sensitive results than the URA methods. Both of the ultrasonic methods show less than 10% deviation from the values obtained in optical microscope.

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