Proceedings of the International Congress on Advances in Applied Physics and Materials Science, Antalya 2011

Examining with the Sintered Temperature of Mean Grain Size of B_4C–Al–Ni Composites by Ultrasonic Techniques

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Hard, tough, lightweight boron–carbide–reactive metal composites, boron–carbide–aluminum composites, are produced. The ultrasonic velocity and attenuation were measured on B_4C–Al–Ni ceramic–metal composites, which are sintered in the temperature range 500–1200 °C, by using the pulse echo method. Ultrasonic velocity, ultrasonic attenuation and rate of screen heights of successive peaks were determined according to the pulse-echo method by using 2 MHz and 4 MHz probes. It was observed that the mean grain size of samples has been changed with the sintering temperature. The velocity was found to be pretty sample dependent at room temperature and increased with increasing sintering temperature. The ultrasonic attenuation at room temperature was found to be more sample dependent. The sintering temperature variation of ultrasonic attenuation exhibited broad minimum values around 1200 °C and sharp maximum values at 500 °C. The increase observed in the temperature variation of longitudinal velocities and attenuation has been qualitatively explained with the help of the temperature variation of decrease value.

PACS: 43.35.Ae, 81.70.–q, 43.35.Zc

1. Introduction

Ultrasonic technique is a nondestructive method in which beams of high-frequency sound waves are introduced into materials for the detection of surface and subsurface flaws in the material [1]. Ultrasonic testing (UT) is a nondestructive method in which beams of high-frequency sound waves are introduced into materials for the detection of both surface and internal flaws. The UT sound waves travel through the material with some attendant loss of energy and are deflected at interfaces and/or defects [2]. 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. The experimental study of the grain size influence on the ultrasonic parameters such as the velocities and the attenuation coefficients in polycrystalline materials requires to take into account the scattering of the ultrasonic waves [3, 4].

Boron carbide is one of the most important non-metallic hard materials with outstanding properties such as extremely high hardness (9.5 Mohs scale), excellent hot strength, remarkable corrosion resistance, very low specific gravity (2.52 g/cm^3) and high elastic modulus [5]. Ceramic materials, which include monolithic ceramics and ceramic–matrix composites, have been identified as potential candidates for high-temperature structural applications because of their high-temperature strength, light weight, and excellent corrosion and wear resistance [6]. This work has shown that ultrasonic velocity, ultrasonic attenuation, and relative amplitude measurements can be used to estimate mean grain size in composites.

2. Theory

In this study we have examined changing with the sintered temperature of mean grain size of B_4C–Al–Ni composites by ultrasonic techniques. We used three ultrasonic methods which are velocity, ultrasonic attenuation, and one similar to ultrasonic relative attenuation (URA) method.

3. Ultrasonic methods

Longitudinal ultrasonic velocities of samples have been measured by a pulse-echo method with a Sonatest Sitescan 150 pulser/receiver instrument. We have used two probes that have different frequencies, 2 MHz probe (Sonatest SLH2-10, T/R) for to plot reference graphs and 4 MHz probe (Sonatest SLH4-10, T/R) to evaluate mean grain size of samples. According to Hirsekorn, in the Rayleigh scattering region, longitudinal and transverse ultrasonic wave velocities have been given as a function of wave number, k, and the grain radius, a. For a longitudinal wave, phase and group velocities are given and experimental work of these relations has been reported in [7]. These formulae have shown us ultrasonic velocity and grain size between relationship. Ultrasonic attenuation due to energy scattered at the grain boundaries depends on the ultrasonic wavelength, the grain size, and certain material properties such as elastic constants. Roney's generalized approach to ultrasonic attenuation is used. In our study, we used this formula for calculating attenuation coefficient as follows:

$$\alpha = \frac{1}{d} 20 \log \frac{A_1}{A_2}\,, \tag{1}$$

where α is the ultrasonic attenuation, d is the thickness of materials, A_1 and A_2 is successive amplitude of reflected ultrasonic wave in materials boundary, respectively [8]. Also we used one of the new methods URA which depends on the first backwall echoes of different samples. 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.

4. Experimental

4.1. Materials

The metal–matrix composites were examined in this investigation. The powders used for the preparation of B_4C–Al–Ni composite samples were 60% B_4C, 10% Al, and 30% Ni. Chemical bath contained nickel chloride ($NiCl_2 \cdot 6H_2O$) from which Ni atoms dissociate or release to bath. The powder sizes were nominally −325 mesh. Powders Al and Ni were etched in hydrazine hydrate acid washed with distilled water and dried. The surface of the investigated powders were sensitized and activated prior to electroless nickel coating.

4.2. Metal–matrix composites fabrication

All the powders were placed in a 15 mm diameter cylinder-shaped steel mold and pressed using a hydraulic press at a pressure of 300 bar. B_4C–Al–Ni composite samples were sintered at the different temperatures ranging from 500 °C to 1200 °C in an argon shroud in Phoenix microwave furnace for 2 h. Sintered samples were characterized using Leo 1430 VP equipped with Röntec energy dispersive X-ray (EDX) model scanning electron microscopy (SEM). SEM images of sintered B_4C–Al–Ni samples are shown in Fig. 1.

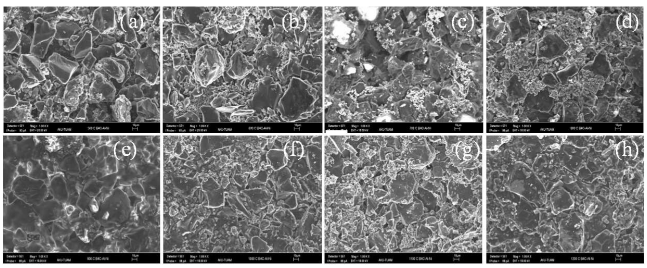

Fig. 1. SEM images of B_4C–Al–Ni composite samples: (a) 500 °C, (b) 600 °C, (c) 700 °C, (d) 800 °C, (e) 900 °C, (f) 1000 °C, (g) 1100 °C, and (h) 1200 °C.

5. Results

Three method and mean grain size of B_4C–Al–Ni composite samples, which are obtained by SEM images, were given in Table I and these samples were calculated by using pulse-echo technique.

According to Table I, ultrasonic velocity, ultrasonic attenuation, and the rate of peak heights–mean grain size reference graphs were plotted and given in Figs. 2, 3, and 4, respectively.

TABLE I

Sintering temperature, ultrasonic velocity, attenuation, rate of peak heights and mean grain size values of samples.

Sintering temperature [°C]	Ultrasonic velocity [m/s]	Attenuation [dB/mm]	Rate of peak heights	Mean grain size [μm]
500 (EP)	1.082	6.34	0.128	10.68
600 (EP)	1.118	5.9	0.1795	12.3
700 (EP)	1.302	4.08	0.3182	13.24
800 (EP)	1.457	3.38	0.347	14.3
900 (EP)	1.507	2.75	0.426	16.9
1000 (EP)	1.559	1.93	0.518	17.5
1100 (EP)	1.578	0.88	0.76	19.1
1200 (EP)	1.669	0.26	0.909	20.4

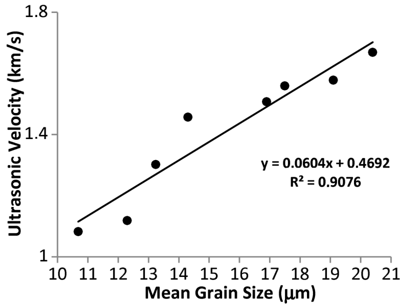

Fig. 2. Ultrasonic velocity–mean grain size graph of B_4C–Al–Ni composite samples.

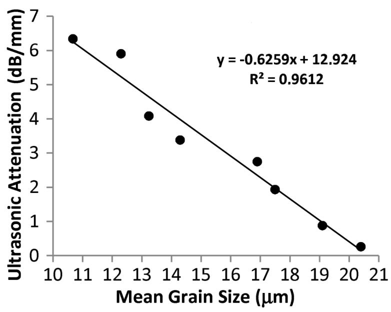

Fig. 3. Ultrasonic attenuation–mean grain size of B_4C–Al–Ni composite samples.

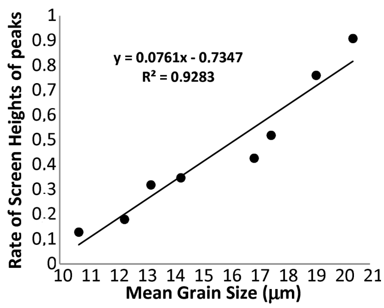

Fig. 4. Rate of screen heights of peaks–mean grain size of B_4C–Al–Ni composite samples.

TABLE II

Evaluated mean grain size of B_4C–Al–Ni composite samples by using 4 MHz probe.

B_4C–Al–Ni		500 °C	600 °C	700 °C	800 °C	900 °C	1000 °C	1100 °C	1200 °C
experimental mean grain size [μm]		10.68	12.3	13.24	14.3	16.9	17.5	19.1	20.4
Velocity	ultrasonic velocities 4 MHz [km/s]	1.118	1.316	1.491	1.686	1.791	2.045	2.363	2.451
	evaluated mean grain size [μm] $y = 0.0604x + 0.4692$	10.74	14.02	16.92	20.15	21.88	26.09	31.35	32.81
	Δ (grain size)	0.06	1.72	3.68	5.85	4.98	8.59	12.25	12.41
Attenuation	ultrasonic attenuation 4 MHz [dB/mm]	6.41	6.04	5.15	4.35	3.33	2.19	1.55	0.86
	evaluated mean grain size [μm] $y = -0.602x + 12.476$	10.08	10.69	12.17	13.50	15.19	17.09	18.15	19.30
	Δ (grain size)	0.60	1.61	1.07	0.80	1.71	0.41	0.95	1.10
URA	rate of screen heights 4 MHz	0.17	0.14	0.20	0.23	0.44	0.47	0.68	0.73
	evaluated mean grain size [μm] $y = 0.0761x - 0.7347$	11.89	11.51	12.32	12.73	15.37	15.88	18.64	19.21
	Δ (grain size)	1.21	0.79	0.92	1.57	1.53	1.62	0.46	1.19

A linear relation has been observed at all of these graphics in Figs. 2, 3, and 4. Mean grain size of B_4C–Al–Ni composite samples were evaluated by using 4 MHz transducer's value and are given in Table II. The mean grain sizes both calculated and measured were plotted in Fig. 5 by using values from Table II.

6. Conclusion

In this study we have investigated relation between ultrasonic velocity, ultrasonic attenuation, the rate of peak heights and mean grain size at different temperature of the studied boron carbide–aluminium–nickel ceramic–metal matrix composite samples. All three methods used in this study gave comparable results as shown in Fig. 5. The two ultrasonic methods give closer results to each other when the grain sizes of samples are absolutely larger. The ultrasonic velocity methods have given more sensible results than the URA methods. Both of the ultrasonic methods show less than 10% deviation from the values obtained in SEM images.

Acknowledgments

The authors are grateful to Afyon Kocatepe University, Technical Application and Research Center for their help in experimental work. This work has been supported by Afyon Kocatepe University research grant number 06.FENED.11.

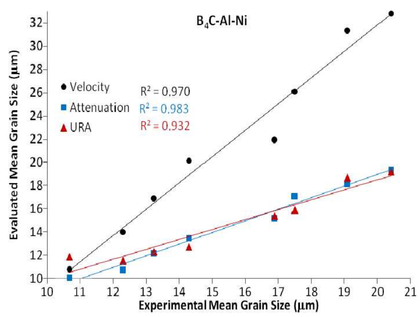

Fig. 5. Comparison of experimental and evaluated mean grain size of B_4C–Al–Ni composites.

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