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EVALUATING OF THE RELATIONSHIPS BETWEEN AVERAGE PARTICLE SIZE AND MICROSTRUCTURE-MECHANICAL PROPERTIES OF MATERIALS PRODUCED IN DIFFERENT COMPOSITIONS USING ULTRASONIC METHOD

Pulse-echo ultrasonic test, which is one of the non-destructive testing methods, was used to measure ultrasonic quantities such as longitudinal velocity (V_L), shear velocity (V_T) and attenuation coefficient (α) in FeCrMn composites. The corresponding elastic constants were determined depending on the longitudinal and transverse velocity. The aim was to reveal the correlation between the microstructural and mechanical properties of FeCrMn composites and ultrasonic quantities. The effect of adding Cr particles on V_L and V_T velocities is obviously attributed to the change in elastic and shear modulus of FeCrMn composites. It was found that both V_L and V_T velocities, Young's modulus (E) and shear modulus (G), as well as hardness values, changed approximately linearly with increasing Cr content. In this study, samples with different volumetric compositions were produced using the powder metallurgy method. It has been revealed that both the applied method and the increase in the amount of Cr have a significant effect on the velocities of V_L and V_T. The increase in V_L and V_T is due to the increase of Cr particles, the homogeneous distribution of Cr, the formation of samples especially at a certain temperature, and the decrease of porosity. As a result of these, a decrease in attenuation values was observed depending on the mean grain size. Elastic constants were found to vary in the same way as ultrasonic velocities. By increasing the Cr content both the hardness values and the shear modulus were improved and a good correlation was observed with the grain size.

Keywords: Non-destructive ultrasonic testing; Ultrasonic velocity and attenuation; powder metallurgy; micro-hardness

1. Introduction

The versatile application of composite materials, which are expressed as the most common composite materials today, is mostly associated with improved properties including ultrasonic, mechanical and physical properties compared to traditional monolithic materials such as metals, ceramics and polymers used as matrix [1-4]. Composite material properties also need to be improved in order to meet the need for materials with different properties for new areas of use with the developing technology [5-7]. The application of composites has become widespread in different industries such as automotive and aerospace where the mechanical and physical properties of the components are important, using ultrasonic properties to obtain information about the internal structure without damaging the material [8-11]. Destructive, semi-destructive and non-destructive testing (DT and NDT) techniques are available for the characterization of a product or material or component, and these characterization techniques are the main tool for quality control and quality assurance. Ultrasonic wave velocity and attenuation are important variables required for ultrasonic material characterization. In addition, ultrasonic wave velocity gives necessary information about the mechanical, anisotropic and elastic properties of the medium through which it passes, since it is related to the elastic constants and density of a material [12-13].

Since ultrasonic characterization has proven that it is possible to evaluate the microstructure and mechanical properties of composite materials by measuring ultrasonic parameters, it has been tried to get the best results by using different production methods to evaluate the properties of composite materials. One of these production methods is powder metallurgy (P/M) technology. It is a more advantageous production technique compared to other production methods in terms of producing advanced technology materials, starting from raw materials in powder form, having a high strength, having a sintering temperature less than the melting point, producing the product at a reasonable low cost,

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less errors and providing the physical and chemical properties of the desired powders [14-16]. Due to these properties, one of the most important ways to improve the properties of composite materials produced by the powder metallurgy method is the changes made with the added additives [17-19]. For the reasons mentioned above, an experimental study was carried out to investigate the relationship between the microstructural and mechanical properties of ultrasonic characterization by measuring and evaluating the ultrasonic parameters of FeCrMn composites reinforced at different rates, and also to investigate the applicability of the ultrasonic pulse-echo technique. FeCrMn samples were produced using powder metallurgy method. The reason for choosing the PM technique is that it offers a significant advantage in directing or influencing microstructural parameters, producing higher quality and more economical (high strength, high hardness and density) materials, and improving poor surface properties, respectively.

2. Experimental procedures

2.1. Materials and powder metallurgy

Samples have been prepared by using powder metallurgy method. Fe, Cr, and Mn powders were used by the powder metallurgy method. In the experiments, iron (Fe) powder of 99.8% purity and grain size less than 200 mesh, chromium (Cr) powder of 99.9% purity and grain size less than 200 mesh, and 99.5% purity were used as metal powders. Manganese (Mn) powder with a grain size of 50 mesh was used. All these powders used in composite production were supplied by Sigma Aldrich Materials. In the study, the samples produced by the powder metallurgy method, volumetric change compositions, and codes given to the samples are given in TABLE 1. The powders taken in determined amounts were mixed in the mixer rotating at 20 *dv*/*dk* speed for 24 hours in order to obtain a homogeneous mixture. The prepared powder mixtures were pressed under a pressure of 305.9 kg/cm^2 in a cylindrical steel mold with a diameter of 30 mm in a uniaxial hydraulic press at room temperature. All samples were sintered in a conventional sintering furnace at 1400°C in an argon atmosphere. The LEO 1430 VP model Scanning Electron Microscope equipped with a RONTEC EDX analyzer was used for microstructural and EDX compositional analysis. Phases in composite samples were detected using Shimadzu XRD-6000 brand X-ray diffraction and CuKα (λ = 1.5406 A°) radiation. Microhardness measurements were made on a Shimadzu HM-2V (Vickers tip) microhardness tester using a 50 g load.

TABLE 1

Chemical composition of the composite materials (wt %)

Composite samples with code	Fe	Cr	Mn
CS-1	96.4%	—	~3.5%
CS-2	89.25%	7.24%	~3.5%
CS-3	82.12%	14.39%	~3.5%
CS-4	75.08%	21.45%	~3.5%
CS-5	68.13%	28.42%	~3.5%

2.2. Ultrasonic measurements

A Sonatest Sitescan 150 pulser/receiver instrument was used to examine the ultrasonic properties of samples produced by the powder metallurgy method. In order to ensure the accuracy of velocity measurements in ultrasonic material characterization studies, the samples were flat and the surfaces were smooth and parallel. The cylindrical shape and dimensions of the samples were determined as (8 mm × 30 mm) in order to avoid the side wall effect during ultrasonic measurements.

In this study, produced by two companies probes that have frequencies 2 MHz probe having 10 mm diameter (Sonatest SLH2 10, T/R) for longitudinal waves and 4 MHz probe having 14,5 mm diameter (GE Inspection Technologies MB 4Y) for transverse waves were used. The ultrasonic velocity was calculated by measuring the samples thickness with a micrometer and dividing twice the samples thickness by the time-of-flight between zero-crossing of two back-wall echoes [20]. Sonatest sonagel-W liquid gel couplant, which can be used on all types of metallic surface, was applied as a coupling medium at the probe-sample interface. The mean velocities were obtained by averaging the three independent measurements. Measurement of ultrasonic wave speed within the material and attenuation of ultrasonic waves are parameters important to its characterization [21]. Attenuation refers to the energy loss associated with the decrease in the ultrasonic wave amplitude due to both scattering and absorption [22]. This effect is seen as a loss of signal amplitude on the screen of an ultrasonic faw detector. Ultrasonic wave loss is usually expressed in logarithmic units such as Neper or dB. The attenuation coefficient (α) was determined based on the ratio of the amplitudes of successive reflected back wall peaks (A_1 and A_2) at the same point within the boundaries of the material by the following equation [23]:

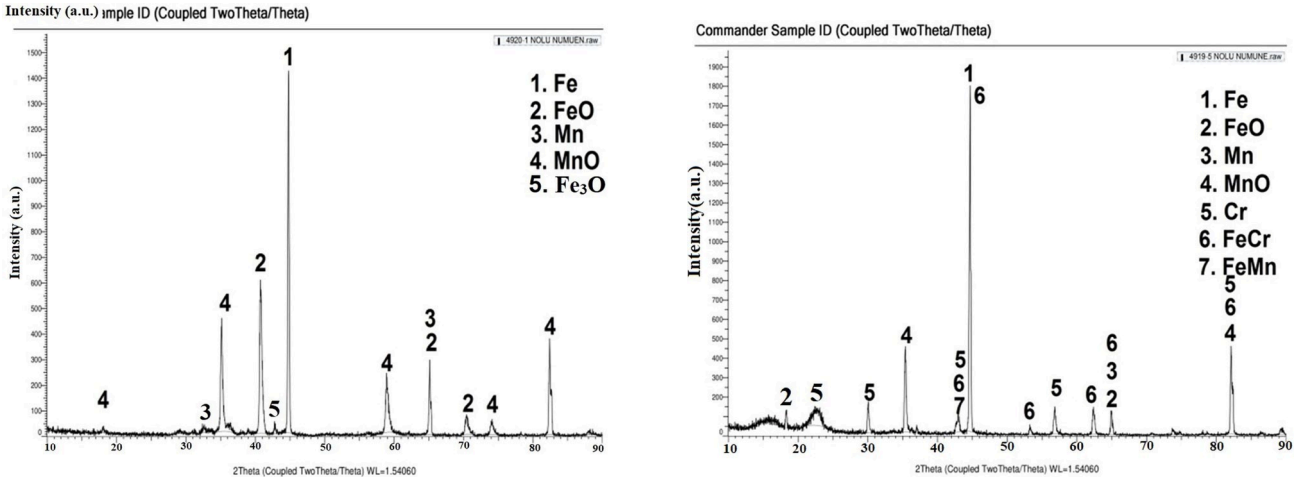
$$Attenuation\ coefficient\left(dB/mm\right)=\frac{1}{d}20\log\frac{A_1}{A_2} \qquad (1)$$

where A_1 and A_2 are the amplitude of two consecutive back wall echoes, d is the thickness of the samples in (mm). Ultrasonic longitudinal and transverse attenuation measurements were repeated three times using consecutive peaks in ultrasonic velocity values, and the mean value was reported with the standard deviation.

The density of the samples was measured using the Archimedes technique. The elastic constants of the produced samples were derived from standard velocity-elasticity relations. The elastic constants of the samples, including the shear modulus (G) and Young's modulus (E) were deduced from the measured ultrasonic longitudinal (V_L) and transverse (V_T) wave velocities and density (ρ) [24] as follows:

$$Young's\ modulus\left(E\right)=\rho V_T^2\frac{3V_L^2-4V_T^2}{V_L^2-V_T^2} \qquad (2)$$

$$Shear\ modulus\left(G\right)=\rho V_T^2 \qquad (3)$$



a) CS-1 b) CS-5

Fig. 1. The X-ray analysis composite samples

3. Results and discussion

3.1. Microstructural hcaracterization of samples

The effects of both the different content of the samples produced in various compositions by powder metallurgy method and the variation in grain sizes as a result of their interaction with each other on the microstructure are shown in Fig. 1. Analysis of samples CS-1 and CS-5 can be seen from the produced samples. Chromium powders were not used in sample CS-1. Fe, FeO, Mn and MnO phases formed in sample CS-1 were determined. The phases determined in sample CS-1 were obtained with low hardness phases. In the X-ray analysis of sample CS-5, Fe, FeO Mn, MnO, Cr, FeCr and FeMn phases were determined. The presence of Cr powders in the CS-5 sample provided a harder sample. SEM images of sample CS-5 show that there is good sintering.

The effects of both the different content of the samples produced in various compositions by powder metallurgy method and the variation in grain sizes as a result of their interaction with each other on the microstructure are shown in Fig. 2.

In Fig. 2, SEM images of the samples produced by the powder metallurgy method are given. It is seen that the absence of Cr powder in sample CS-1 is also reflected in the microstructure. Although there is grain coarsening in the microstructure, it is observed that the porous structure and microcracks are formed. It was determined that the porosity decreased with the increase of Cr ratio in CS-2, CS-3 and CS-4 samples. It is seen that the CS-5 sample with the highest Cr ratio has the least porosity. The hardness values also support the reduction of porosity.

3.2. Ultrasonic measurements

In this study, it is aimed to show whether the ultrasonic and mechanical properties of composite materials produced by powder metallurgy method depend on grain size. The numerically calculated and measured values of ultrasonic and mechanical properties of samples prepared in different compositions are presented in TABLE 2. The variation of ultrasonic and mechanical properties of Fe-Cr-Mn samples produced by powder metallurgy method with mean grain size is shown in Fig. 3. As seen in Fig. 3a and Fig. 3b, the ultrasonic velocity in all samples increases with an increase in Cr content. For instance, the velocity of longitudinal wave increased from 3734 to 4853 m/s when Cr amount increases from 0% (sample CS-1) to 28.42% (sample CS-5). The addition of Cr at different rates significantly affects both the longitudinal and transverse velocities. Velocities of V_L and V_T are higher in the presence of 28.42% (sample CS-5) compared to 0% (sample CS-1) as indicated in TABLE 2.

The ultrasonic wave travels faster in the harder phase (FeCr and FeMn) compared to the FeO and MnO phase. Fig. 3 shows that the mean grain size, ultrasonic longitudinal and transverse

TABLE 2

Ultrasonic and mechanical properties composite samples

Composite Samples	Mean Grain Size (μm)	V_L (m/s)	V_T (m/s)	Longitudinal Attenuation (dB/mm)	Transverse Attenuation (dB/mm)	Elastic Modulus (GPa)	Shear Modulus (GPa)	Micro-hardness (0.05 HV)	Density (g/cm^3)
CS-1	7.50	3734 ± 63	1731 ± 54	0.44 ± 0.02	0.75 ± 0.03	39.93	14.65	138.6	4,88
CS-2	8.56	3880 ± 17	1852 ± 53	0.33 ± 0.03	0.57 ± 0.03	46.27	17.11	148.6	4,99
CS-3	13.87	4439 ± 45	1958 ± 40	0.25 ± 0.01	0.45 ± 0.03	56.85	20.61	162.9	5,38
CS-4	16.76	4730 ± 98	2146 ± 74	0.14 ± 0.01	0.33 ± 0.01	69.88	25.49	184.3	5,54
CS-5	30.30	4853 ± 52	2855 ± 39	0.07 ± 0.01	0.27 ± 0.01	116.63	47.19	206.5	5,79

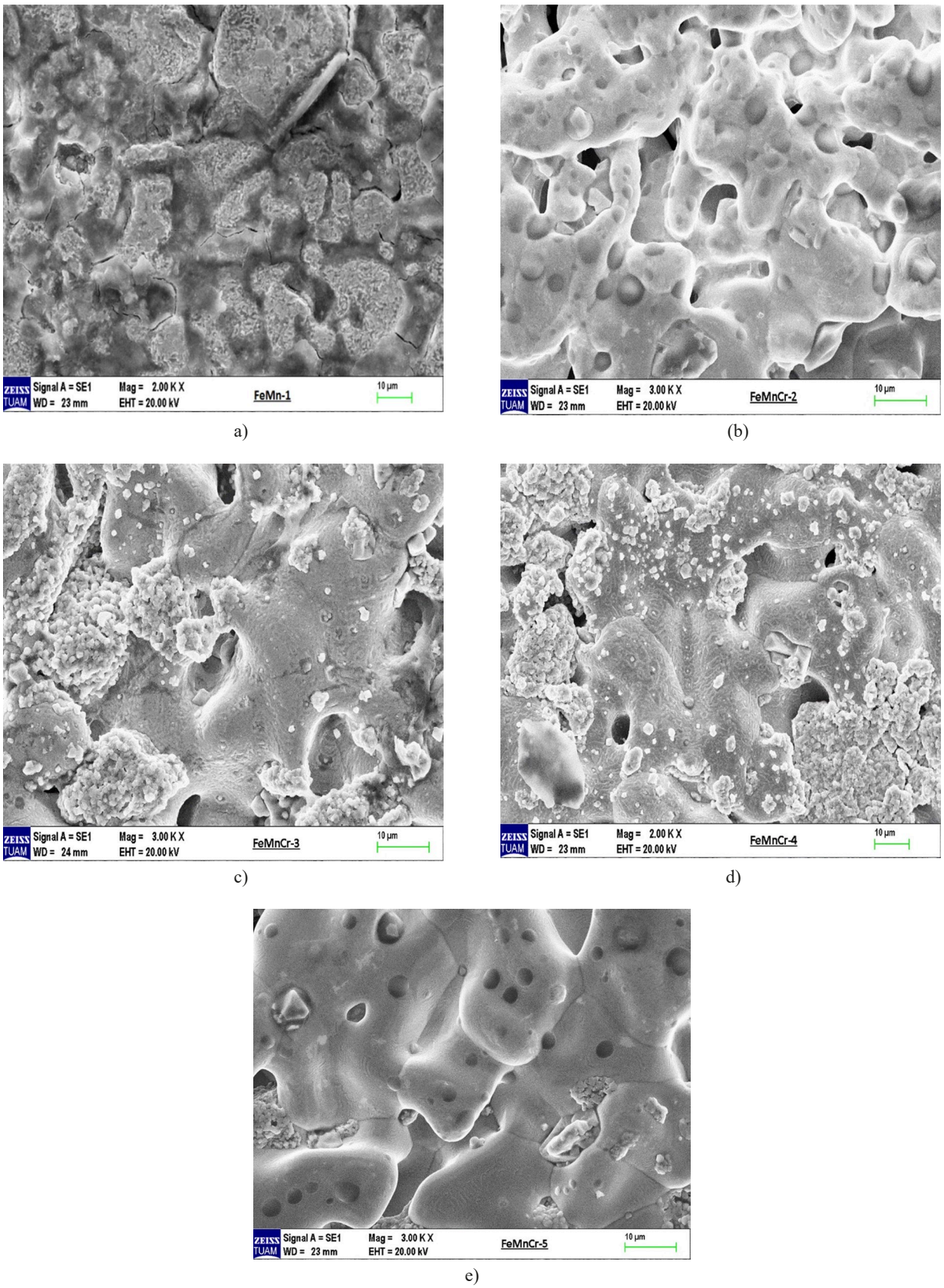
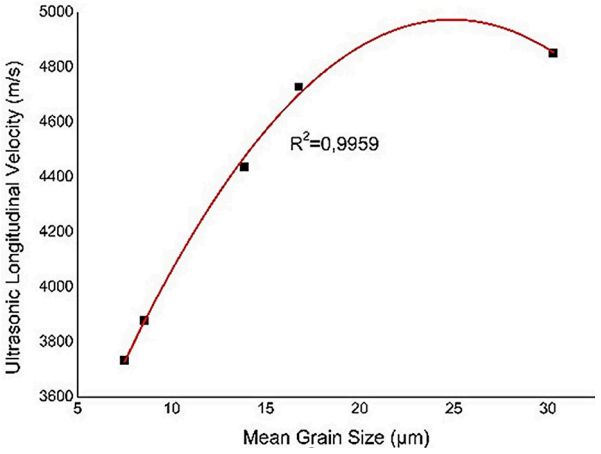


Fig. 2. The SEM analysis of composite samples with a) CS-1, b) CS-2, c) CS-3, d) CS-4 and e) CS-5

velocity values, elastic and shear modulus, density and hardness values of sintered Fe-Mn and Fe-Cr-Mn samples increase linearly. In Fe-Cr-Mn composites, the values of V_L and V_T velocities increase with increasing Cr content in the presence of larger Cr content, as expected from a harder matrix. Considering the attenuation values, the results are as expected. According to the measurements taken, it is seen that the attenuation values decrease as the mean grain size increases (Fig. 3c

and Fig. 3d). As the mean grain size increases, the absorption process decreases. Similar to velocity measurements, the main contributions to the attenuation of the ultrasonic wave are the FeCrMn content, the uniformity of distribution in the structure, and the amount of porosity. It is also seen that the attenuation values depend on the frequency of the ultrasonic wave and depend on the microstructure of the sample. When an ultrasonic wave travels through the sample and encounters FeCrMn of different composition, the ultrasonic wave energy is re-emitted by FeCrMn particles, which act as an oscillator. A large content of FeCrMn particles causes more attenuation of the ultrasonic incident wave, thus resulting in higher attenuation values. The change in attenuation value can be associated not only with the presence of different amounts of FeCrMn particles, but also with the degree of dispersion homogeneity of FeCrMn. This decrease in the absorption process is seen as a decrease in the amount of attenuation with increasing grain size. A homogeneous distribution of FeCrMn particles reduces the interparticle gap and increases the probability of interaction of the moving ultrasonic wave with the FeCrMn particles and the surrounding increased Cr matrix, resulting in an increase in the attenuation of the ultrasonic wave. The increase in the amount of Cr and the reaction of the grains with each other at a certain temperature, that is, the increase in grain size reduces the porosity. It supports the results obtained in SEM images and XRD analysis. The transmitted signal is lost due to reduced porosity and increased grain size. This is seen as a decrease in attenuation values.

As can be seen in TABLE 2, elastic constants change in the same way as ultrasonic velocities. Young's modulus and Shear's modulus were calculated using the obtained wave velocity and density values. Primarily, the E and G values obtained from ultrasonic measurements increase in the same way as the mean grain size values obtained from SEM images (TABLE 2). Experimental E and G values of FeCrMn samples increase with increasing Cr content. Higher E values in FeCrMn samples generally suggest an effective interfacial bond between Fe, Cr and Mn particles, thanks to its homogeneous distribution and smaller interparticle spacing. The distribution of Cr particles in the structure increases the charge transfer from the matrix to the reinforcement and offers higher E and G values [25-26]. The correlation coefficient (R^2) of the fitted line in the graph given is close to 1, which indicates that there is a linear relationship (Fig. 3e and Fig. 3f). Also, the higher the Elasticity or Shear modulus of a medium, the higher its fracture strength under pressure [27]. The hardness of the base material is between 138.6 $HV_{0.05}$ and 206.5 $HV_{0.05}$, depending on the amount of Cr, it contains. Hardness increases with increasing amount of Cr. Hardness values vary depending on the phases obtained. Since there is FeO and MnO phase in sample CS-1, the hardness is low. In addition, CS-1 sample does not contain Cr, so its hardness



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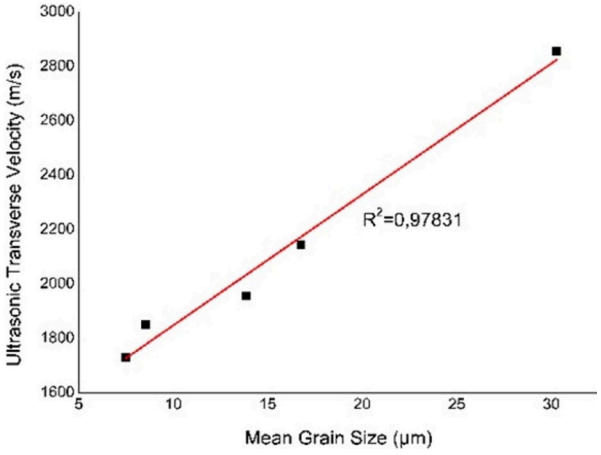

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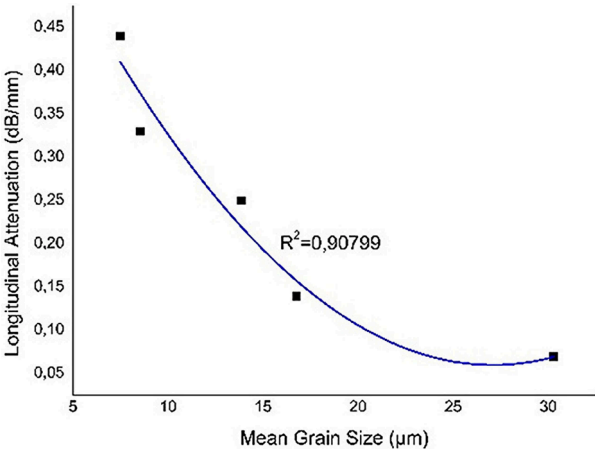

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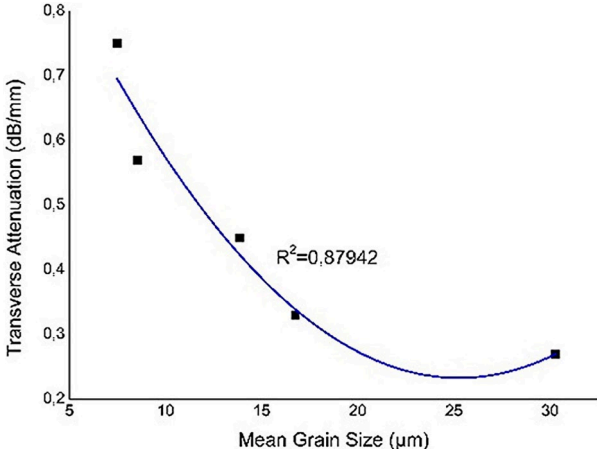

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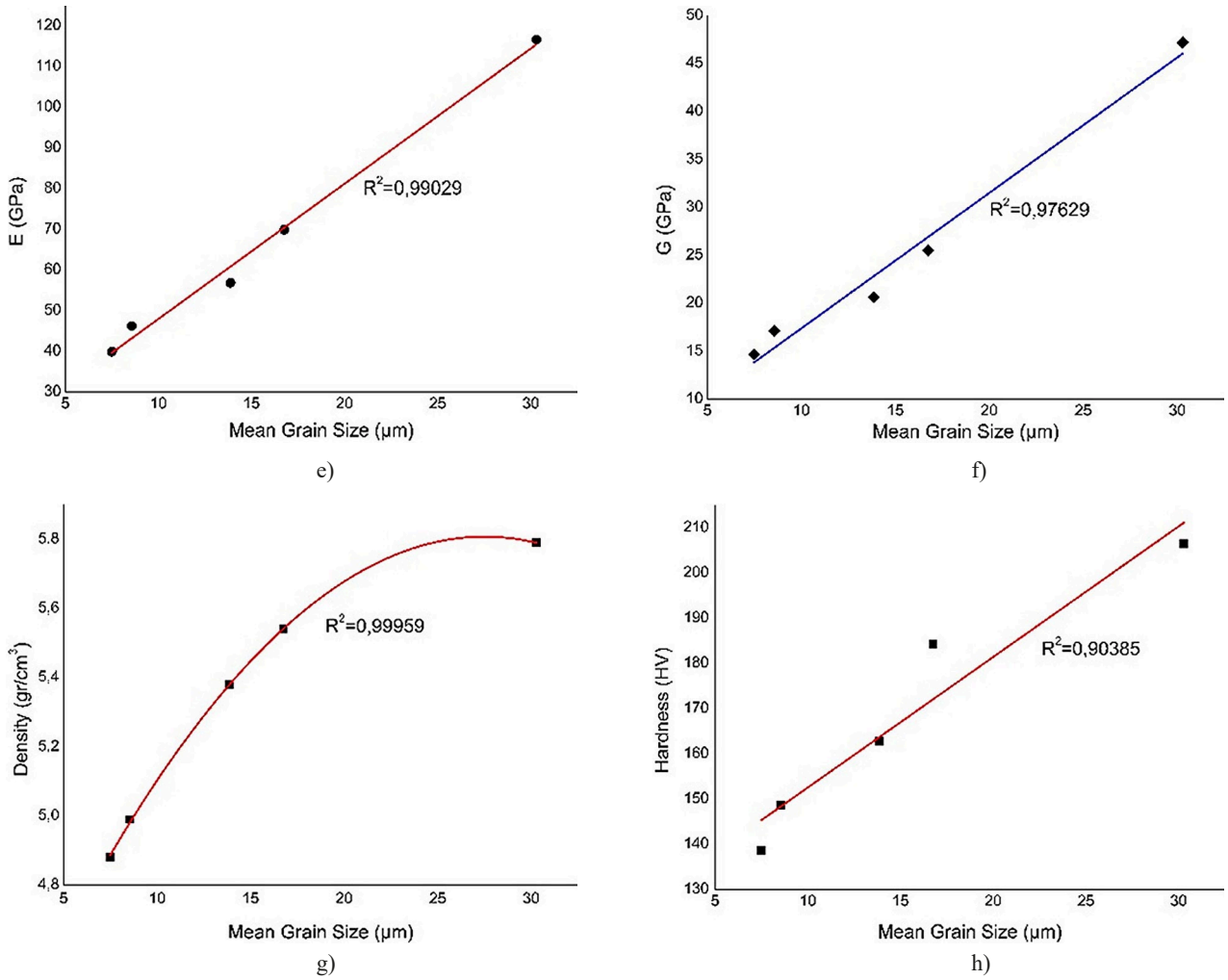

Fig. 3. The relationship between mean grain size (µm) values and the ultrasonic-mechanical properties of composite samples

value is lower than other samples. With the increase of Cr ratio in CS-2, CS-3, CS-4 and CS-5 samples, the presence of existing FeCr, Cr and FeCr phases significantly increases the hardness value (Fig. 3h) [28-30].

Density changes as a function of FeCrMn content are given in TABLE 2, and the density-mean grain size change curve is given in Fig. 3g. As the particle size increases, the density of FeCrMn samples prepared at different volume ratios increases. It is also known that the sintering temperature has an effect on the density of the material. The highest sintered density value was calculated as 5.79 g/cm^3 for the composite samples sintered at 1400°C for 2 hours. It is believed that temperature plays an important role in the diffusion of air in the cold pressed sample and the contact of the powders with each other during sintering. Accordingly, the increase in the amount of Cr as the binder phase increases the contact of the particles with each other and allows them to stick, and causes the density to increase by closing the pores between the particles. The density of the samples increases with increasing mean grain size. The resulting curve shows a nearly linear change.

As two main parameters important for determining the mechanical and ultrasonic properties of a material, the relationship between elastic modulus and hardness is critical for material characterization. Many studies have been conducted to explain the relationship of hardness with elastic modulus [31-32] and shear modulus [33-34], respectively. It can be seen in Fig. 4 that the shear modulus shows a good linear dependence with Vickers hardness for composite samples. The bonds, orientations and interactions between atoms and molecules in a structure become clear with the Young's modulus. If the material is to be

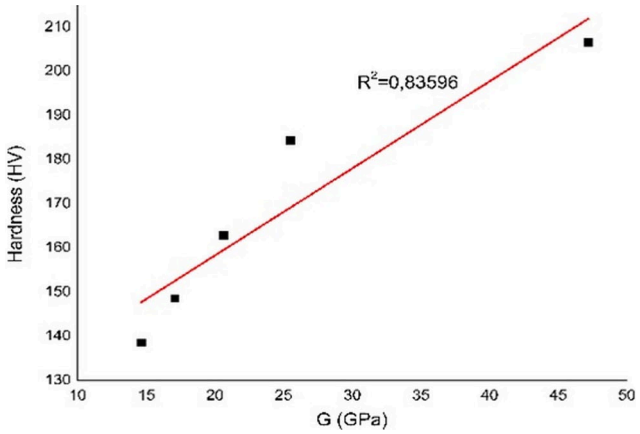

Fig. 4. Variation in microhardness with ultrasonic shear modulus of Fe–Cr–Mn samples

strong, the Young's modulus must be high, which means that the interatomic bonds are stronger and more stable. At the same time, shear modulus, which expresses the strength of a material under shear deformation, which is explained by the dislocation theory [35], is closely related to hardness. In fact, hardness results from deformation associated with the formation and movement of dislocations, which may be more easily induced by shear deformation rather than volume change [33]. Young's modulus (see Eq. (2)), also known as a combination of ultrasonic properties bulk and shear modulus, exhibits a good linear correlation with Vickers hardness [36-40]. In short, considering the relationship between hardness and shear modulus in Fig. 4, we can say that the higher the shear modulus of a material, the more difficult it is for that material to deform, that is, it is more rigid and more prone to maintain its original state. The hardness and shear modulus values of the samples increase with the increase in the amount of Cr.

4. Conclusions

Variations of ultrasonic velocities, ultrasonic attenuation, elastic modulus, shear modulus, hardness and density were correlated with the microstructural properties of FeCrMn samples.

Microstructural features are the FeCrMn content prepared in different compositions, the effect of increasing Cr matrix on grain size, the distribution homogeneity of Fe, Cr and Mn powders. The following conclusion can be reached:

1. In the X-ray analysis of sample FeCrMn, Cr, FeMn, and FeCr phases were determined. The presence of FeMn and FeCr phases obtained in the X-ray analysis of sample CS-5 significantly increased the hardness value.
2. Both V_L and V_T ultrasonic wave velocities increase linearly with the mean grain size due to the increase in Cr content and the homogeneous dispersion of the Cr particles. The increase in V_L and V_T velocities is mainly attributed to the increase in elastic modulus and shear modulus of FeCrMn samples. The velocities of both V_L and V_T vary approximately linearly with the values of E and G. At the same time, when the correlation values between the average grain sizes in the samples and the elastic (E) and shear (G) modulus are examined, a linear relationship is seen more clearly between the numerical values obtained by the effect of the amount of chromium in different proportions prepared by the powder metallurgy method.
3. In FeCrMn samples, the degree of attenuation of the ultrasonic wave is proportional to the uniformity of distribution of the Cr particles, the content of the sample, as well as the ultrasonic wave velocity. There is good agreement between ultrasonic attenuation and mean grain size. The attenuation values decrease linearly with increasing mean grain size. This reduction in attenuation can be attributed to the increase in grain size with increasing Cr content. The ultrasonic signal is lost due to this particle increase in the structure.
4. When the density values of composite samples are examined, the spreading of the binder Cr phase among the powders and the contact of the powders with each other during sintering play an important role in increasing the density.
5. The relationship between hardness and shear modulus for composite samples was investigated and it was seen that they were correlated with each other. Shear modulus is known as a measure of the solid or rigid structure of different types of solid materials and has been found to be quite effective in mechanical strength.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

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