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Elastic Properties of Cr-doped Mn Ferrite



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Abstract

Background: The mechanical properties of Mn-Cr ferrite of different compositions are important for industrial applications in everyday life. Ferrites are used in electric choke coils, electric transformers, and many other electronic and optical devices. The elastic properties can be understood by studying the longitudinal sound velocity (V_l) and shear velocity (V_s).

Results: The longitudinal sound velocity (V_l) and shear velocity (V_s) are measured at room temperature by pulse transmission technique at the frequency of 1659 Hz, which is ideally representing the mechanical properties of the ferrite crystal. The shear sound velocity was found to be around 4×10^3 m/s while the longitudinal sound velocity was ranging according to the doping concentration from 6×10^3 to 1×10^4 m/s. The behavior of Young's modulus (E), rigidity modulus (n), mean sound velocity (V_m), and Debye temperature (θ_D) is quite similar, where a change in the slope was noticed at Cr concentration higher than 0.7 in the spinel lattice.

Conclusion: We have characterized the elastic properties of the spinel structure Mn-Cr ferrite with different Cr ion concentration at a fixed sound frequency using the pulse transmission technique. The longitudinal sound velocity showed an increase with increasing Cr ion concentration, the rigidity constant as well as Debye temperature, Isotropic compressibility, and the acoustic impedance showed behavioral change at Cr ion concentration $x = 0.7$ which is correlated to the intra-ionic distances change due to the replacement of Fe ions with smaller size Cr ions.

Keywords: Mn-Cr ferrite, Mechanical properties, Debye temperature, Acoustic impedance

Introduction

Ferrites have been proven as important compounds that have a wide range of applications in the industry (Abdellatif et al., 2018; Abdellatif et al., 2017), research (Liakos et al., 2016; Abdellatif et al., 2015), physics (Abdellatif et al., 2018; Abdellatif et al., 2015; Abdellatif et al., 2012; Abdellatif et al., 2011), and chemistry of everyday life (Abdellatif et al., 2017; Abdellatif et al., 2016; Abdellatif et al., 2017; Abdellatif et al., 2018; Abdellatif & Azab, 2018). One of the important features of ferrites is its elastic properties; the importance of elastic properties arises due to the fact that studying mechanical and acoustic can give wide insight on the interatomic and interionic forces in solid and crystalline material (Modi et al., 2014; Patange et al., 2013; Modi et al., 2006). The study of elastic properties also helps one to calculate some thermodynamic coordinates such as the Debye temperature. Moreover, the variation of the elastic properties with gradually changing compositions is also important. Previous studies on Cu-

Zn (Modi et al., 2006) and Ni-Zn (Rajesh Babu & Tatarchuk, 2018) show a linear variation of the elastic moduli with concentration. Young's modulus, the rigidity constant, the mean sound velocity, the transverse sound velocity, and the longitudinal sound velocity are known as the elastic parameters of solid crystals. Those parameters can be used to interpret the behavior of the crystalline solid on the basis of binding energy between atoms. Moreover, the Debye temperature can be calculated based on the mechanical parameters using the Anderson formula (Lakhani & Modi, 2010).

One more point, the elastic properties are important in the industry due to the direct relation to the strength of the material under various conditions. The ultrasonic pulse transmission technique is based on transmitting an acoustic pulse at one end of the solid and measuring the attenuation at the other end; accordingly, various mechanical parameters can be calculated. In this work, a systematic study of Mn-Cr ferrite of different compos-

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ition ($x = 0.2, 0.4, 0.7, 0.9, 1$) is undertaken at room temperature. We have paid high attention to calculate the thermodynamical parameters and discuss their correlation with the ferrite structure, and the results are presented in this paper.

Experimental details

Mn-Cr mixed ferrites of the chemical formula $\text{MnCr}_x\text{Fe}_{2-x}\text{O}_4$ where $x = 0.2, 0.4, 0.7, 0.9, 1$ have been prepared by double sintering technique from high purity oxides. Namely, the oxides are manganese(II) oxide (MnO), iron(III) oxide (Fe_2O_3), and chromium(III) oxide (Cr_2O_3), all bought from Sigma-Aldrich. The oxides are mixed together in a stoichiometric ratio to obtain materials with general formula $\text{MnCr}_x\text{Fe}_{2-x}\text{O}_4$. The mixed oxides were continuously grounded for 12 h in a ball miller model PM100 (Retsch, Germany). The oxide's powder was compressed into discs of 10 mm diameter and 3 mm thickness with the help of a home-made uniaxial press, by using an appropriate steel mold. The samples were pre-sintered at 800°C for 8 h. Then, they were grinded to a fine powder and then pressed again into pellets of a rectangular form of $0.8\text{ cm} \times 0.6\text{ cm}$ dimensions using a uniaxial press of pressure $3 \times 10^5\text{ N/m}^2$, and the pellets are sintered at 1200°C for 10 h with a heating rate of 4 deg/min. More details of this method are given in the earlier publication (Ahmed et al., 2012). The elastic properties have been measured by pulse transmission technique (Kohlhauser & Hellmich, 2013; Yu-Huai et al., 1985). In this method, a short pulse from a pulse generator is subjected to the sample through a copper rod using a PZT crystal (Piezoelectric Transducer), which is converted into the mechanical wave and on its arrival at the receiving point, and is converted back into an electrical signal, which is then amplified and fed to an oscilloscope. By knowing the dimensions of the sample and phase difference between the transmitted and received pulse, the sound velocity could be obtained. The sample is housed in a suitable sample holder made of copper rod. Calibration has been done to correct the delay of the sound wave at endpoints. The elastic parameter has been calculated using the following formulas (Venudhar & Mohan, 2002; Ravinder et al., 2001):

$$E = 2n(1 + \sigma) \quad (1)$$

where n is the rigidity modulus calculated from $n = \rho V_s^2$ where ρ is the density of the sample calculated by knowing the mass and dimensions of the sample, V_s is the shear velocity, and σ is the Poisson ratio.

$$\sigma = \frac{(V_\ell^2 - 2V_s^2)}{2(V_\ell^2 - V_s^2)} \quad (2)$$

Debye temperature θ_D as given by Anderson's formula (Venudhar & Mohan, 2002; Ravinder et al., 2001):

$$\theta_D = \left(\frac{V_m h}{k}\right) \left(\frac{3qN\rho}{4\pi M}\right)^{1/3} \quad (3)$$

$$V_m = 1/3 \left(\frac{2}{V_s^3} + \frac{1}{V_\ell^3}\right)^{-1/3} \quad (4)$$

where h is the Planck constant, q is the number of atoms per molecule, M is the molecular weight, and V_m is the average sound velocity.

Results

Figure 1 shows the shear and longitudinal sound velocity respectively as a function of Cr ion concentration at room temperature. It can be seen that the general trend is the increase of the sound velocity with increasing Cr^{3+} ion concentration.

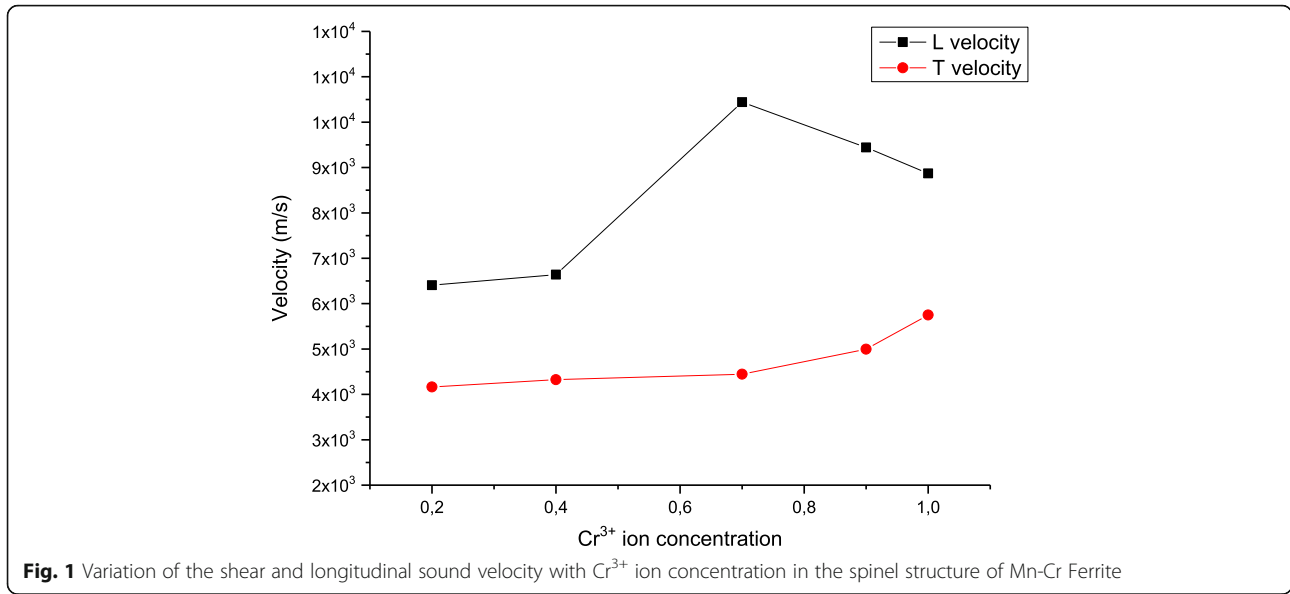
Figure 2 a and b show the rigidity and Young's modulus, respectively. It is clear from the figure that Young's modulus increases with increasing Cr^{3+} ion concentration. The graph could be noted by two straight lines that intersect at $x = 0.7$, which is the point at which the strain decreases by a value corresponding to the decrease in the mean free path of the oscillation.

Figure 3 shows the variation of one of the most important parameters of the thermodynamical coordinate, and it is the Debye temperature of the samples for all mixed ferrites versus average sound velocity. The figure shows that the Debye temperature varies linearly with the average sound velocity V_m . The Debye temperature θ_D has been calculated from the Anderson formula.

Figure 4 illustrates the isotropic compressibility of the sound wave and the acoustic impedance of the specimen for the sound wave. The figure shows that the concentration at $x = 0.7$ has minimum isotropic compressibility and maximum acoustic impedance.

Figure 5 correlates the continuous decrease in the mean atomic weight with increasing Cr^{3+} ion (51.996) concentration due to the fact that Cr ions replace Fe ions (55.847) in the spinel lattice, which is understood as a decrease in the inertia of the molecules of the spinel lattice in which it gets lighter due to ion Cr and Fe ion replacement.

Table 1 shows the variation of density (ρ) and longitudinal (V_ℓ), shear (V_s), and average sound velocity (V_m) with increasing Cr^{3+} ion concentration; the trend of the acoustic and thermodynamical parameters can be followed in the table during the discussion.



Discussion

The mechanical properties of spinel ferrite can be fully understood by considering the interatomic distance of the spinel lattice. It is well known that the ionic radius of Fe³⁺ (0.645 Å) is greater than Cr³⁺ (0.615 Å) so that as the Cr³⁺ content increases, the interionic distance decreases. As the bond strength increases Young’s modulus, the rigidity modulus increases. In other words, one can understand from the experimental data that the binding between the ions of the investigated ferrite is increased successively with increasing Cr³⁺ ion content (Mazen & Elmosalami, 2011; Bartůněk et al., 2018; Sattar & Rahman, 2003) which greatly affects the shear and longitudinal sound velocity in the ferrite slab as shown in Fig. 1. The figure shows an increase in the sound velocity with increasing Cr³⁺ ion concentration. The longitudinal velocity shows a hump at x = 0.7. The Cr³⁺ ion is known to have a strong preference to the octahedral site, which replaces Fe³⁺ content, refer to Table 1 for exact calculated values. We can fully understand the change of the sound velocity by studying its thermodynamical coordinates, since the response of the solid to the acoustic pulse depends on its interatomic arrangement, in a spinel structure, and there are two main interstitial sites, the octahedral (B) and tetrahedral site (A). The vibrating force constant then depends on the ion distribution in the crystal lattice; with the help of Waldron’s method (Mazen & Abu-Elsaad, 2012), the force constant can be calculated using the following equations (Mazen & Abu-Elsaad, 2012):

$$K_t = 7.62 \times M_a \times v_1^2 \text{ N/m} \tag{5}$$

$$K_o = 10.62 \times M_b \times v_2^2 \text{ N/m} \tag{6}$$

where v₁ and v₂ are determined from the IR spectra, then M_a and M_b are the molecular weights of cations in sites A and B, respectively, according to the cation distribution predicted from X-ray. Moreover, the Debye temperature θ_d can be calculated using the following formula (Mazen & Abu-Elsaad, 2012):

$$\theta_D = \frac{hcV_{ab}}{k}, V_{ab} = \frac{V_a + V_b}{2} \tag{7}$$

where V_{ab} is the average wave number of the absorption band in the FTIR spectrum, h is the Planck constant, and k is the Boltzmann constant.

The bulk modulus (B) of the solid is defined by as seen in Eq. 8 (Kittel, 2005):

$$B = \frac{1}{3}[C_{11} + 2C_{12}] \tag{8}$$

where C₁₁ and C₁₂ are the components of the elasticity tensor, for isotropic materials with cubic symmetry like spinel ferrites and garnets; C₁₁ is almost equal to C₁₂ that leads to B = C₁₁.

Hence, the force constant k_{av} = aC₁₁, where k_{av} is the average force constant $K_{av} = \frac{(K_t + K_o)}{2}$.

The longitudinal elastic wave velocity and transverse wave velocity can be calculated from (Kittel, 2005):

$$V_l = (C_{11}/\Delta x)^{1/2}, V_t = V_l/\sqrt{3} \tag{9}$$

However, the mean elastic wave velocity is defined by:

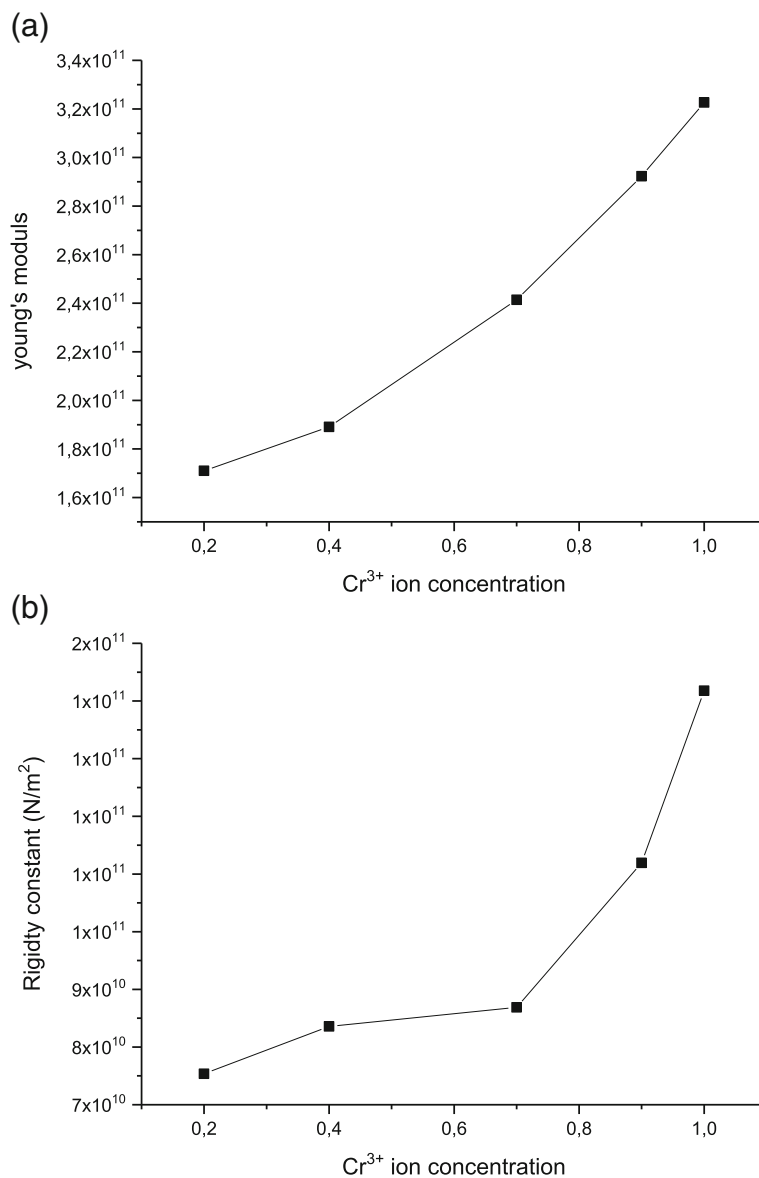


Fig. 2 a Variation of Young's modulus with varying Cr³⁺ ion concentration. **b** Rigidity constant with varying Cr³⁺ ion concentration

$$V_m = \frac{1}{3} \left[\frac{2}{V_l^2} + \frac{1}{V_t^2} \right]^{-1/3} \tag{10}$$

$$E = \frac{1}{2G} \tag{13}$$

Rigidity modulus G is defined by (Raj et al., 2004):

$$G = \Delta x \cdot V_t^2 \tag{11}$$

Then by using Anderson's formula, the Debye temperature can be calculated using the following equation (Raj et al., 2004):

$$\theta_d = \frac{h}{K} [3N_a/4\pi V_{atomic}]^{1/3} V/m \tag{14}$$

Poisson's ratio is defined by:

$$P = 3B - \frac{2G}{6B} + 2G \tag{12}$$

where V_{atomic} is the mean atomic volume, $V_{atomic} = (M/\Delta x)/q$, M is the molecular weight, N_a is Avogadro's number, and q is the number of atoms in the formula unit. However, the importance of elastic properties in spinel structure

In that sense Young's modulus will be defined as:

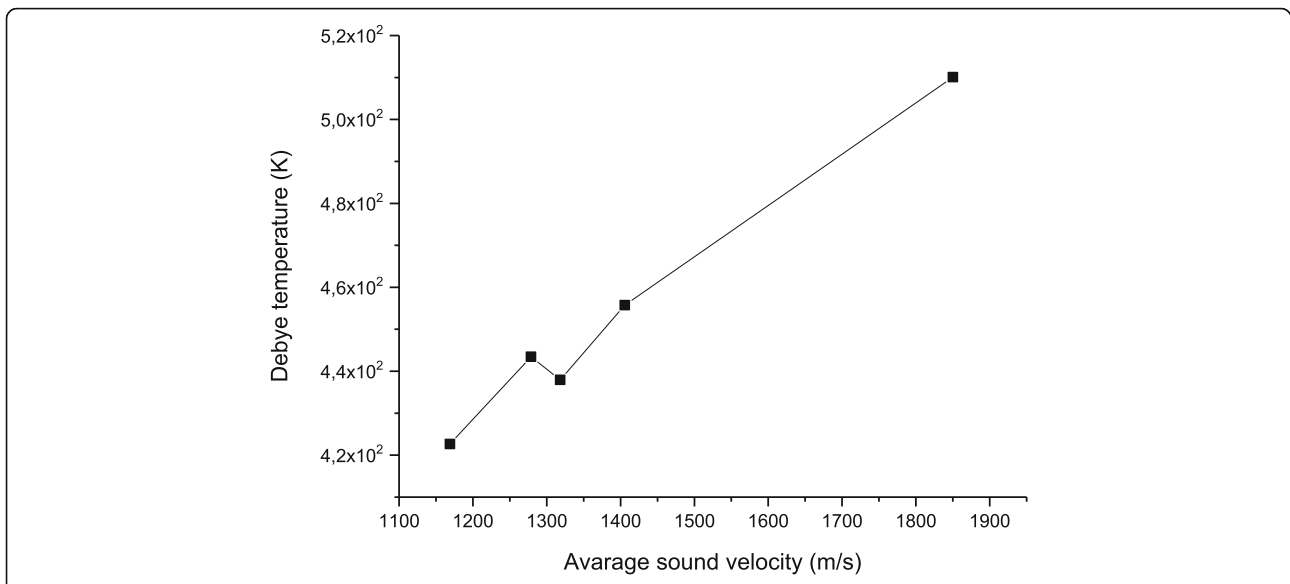


Fig. 3 Variation of the Debye temperature with varying average sound velocity

and all isotropic crystals is the possibility to that it can define the behavior of the binding energies in the crystal lattice. In general, the relation between the average sound velocity increases linearly with the Debye temperature, which is the relation between acoustic parameters defined by sound velocities in the material and the thermodynamic parameters defined by the Debye temperature. However, since ferrite materials are mostly porous which can affect the measured parameters, the velocity should be corrected to zero porosity using the Mackenzie formula (Showry & Murthy, 1991). The hardness of the material can be determined by the Poisson ratio. It is found that

material with a low value of Poisson ratio less than 0.25 is brittle, and those with high value are ductile.

The change in the binding energy due to replacement of Fe by Cr ions also has its effect on the rigidity and Young's modulus shown in Fig. 2; however, a change in the slope can be noticed at $x = 0.7$. This can be understood as the point at which the strain decreases by a value that corresponds to the decrease in the mean free path of the oscillation. The same trend was obtained, where the rigidity modulus increases slightly up to $x = 0.7$, after which it increases suddenly up to $x = 1$. That can be understood as the interionic space changes with changing ion concentration that has a critical value at

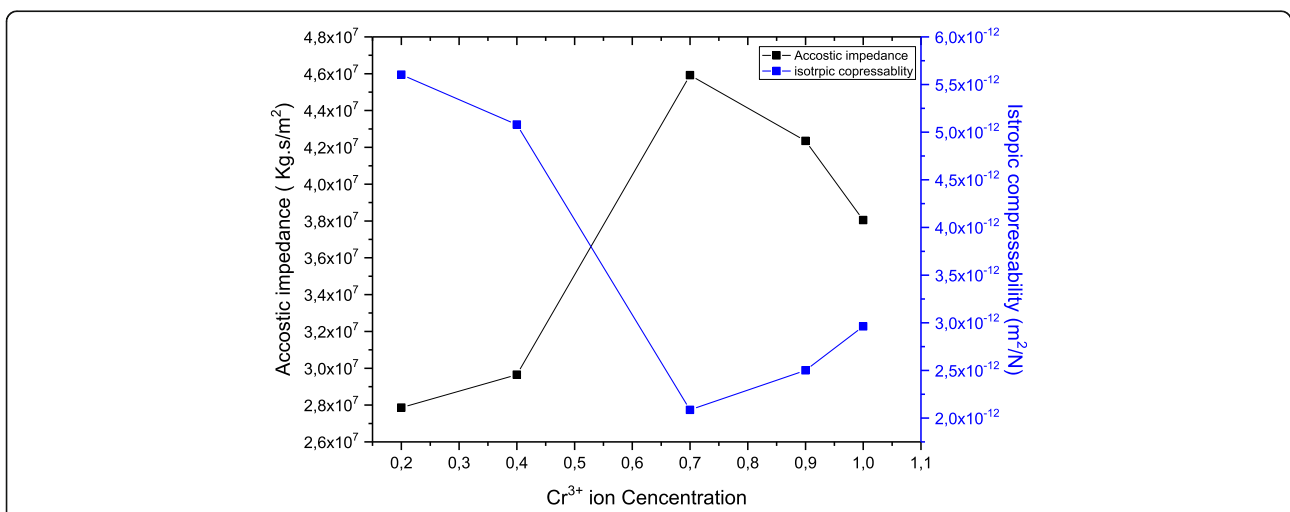
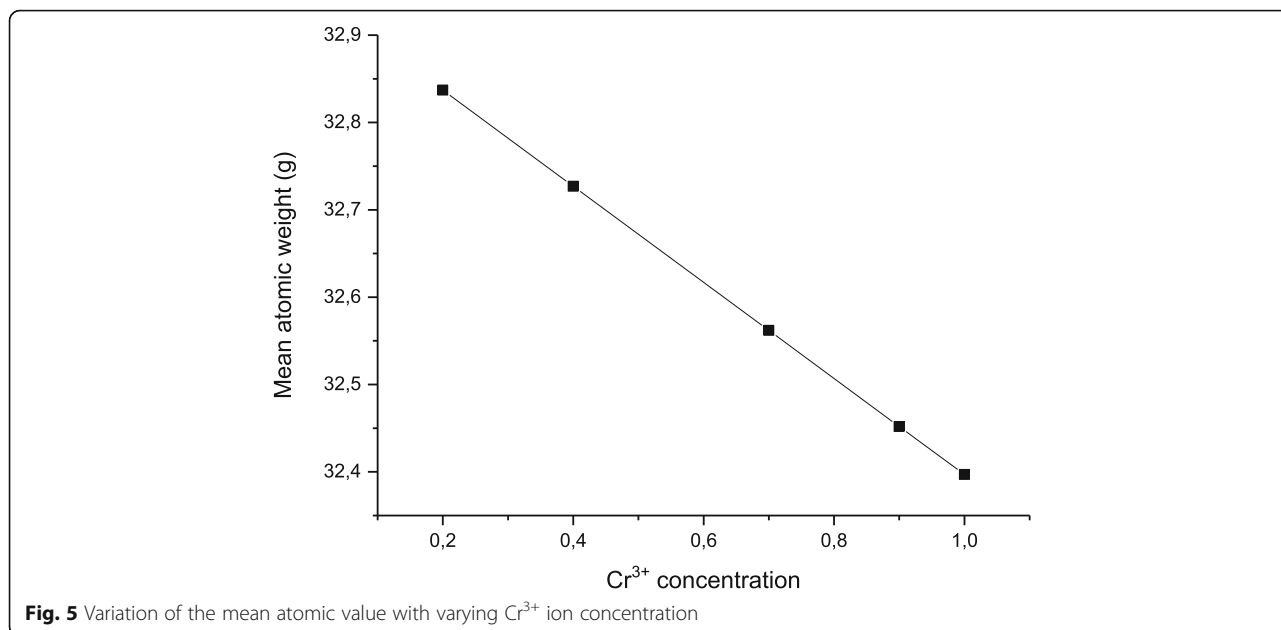


Fig. 4 Variation of the isotropic compressibility and acoustic impedance with varying Cr³⁺ ion concentration in Mn-Cr spinel ferrite



$x = 0.7$. The data in Fig. 1 enhances our expectation where a hump is obtained at $x = 0.7$. The highest normal mode of vibration for the crystal can be found considering the Debye temperature which is shown in Fig. 3. The Debye temperature defines the thermodynamical coordinates of all mixed ferrites versus average sound velocity. It is found that in our samples, the Debye temperature varies linearly with the average sound velocity V_m . The Debye temperature θ_D has been calculated from the Anderson formula. The linear increase of the Debye temperature corresponds to the linear increase of the normal vibrational mode of the crystal, which is correlated to the interatomic distances due to the replacement of Fe ions by Cr ions in the spinel lattice.

The controlling principles of ultrasonic are associated with material properties such as density and modulus of elasticity. In general, there are two types of ultrasonic waves based on material, the bulk waves and guided waves. The bulk waves propagate inside the material while the guided wave propagates near

the surface or along with the interface. The longitudinal waves are those with the displacement in the same direction as travel direction while shear waves are based on the displacement of the amplitude in a direction perpendicular to the propagation direction. According to a theoretical model of attenuation, ultrasonic waves in polycrystalline materials are mainly attenuated by scattering at structure boundaries, grains, grain boundaries, and inclusions. The number of scattering is proportional to the grain volume while the attenuation is a function of the grain size and frequency of the wave. The attenuation coefficient α is a sum of the individual coefficient for scattering and absorption over all frequencies (Mazen & Elmosalami, 2011; Bartůnek et al., 2018; Sattar & Rahman, 2003; Muralidhar et al., 1992; Srinivas Rao et al., 2002; Ravinder & Reddy, 2003).

The attenuation coefficient can be defined as the logarithmic decrement between two consecutive pulse echoes (Lakhani & Modi, 2010; Yu-Huai et al., 1985):

Table 1 Variation of density (ρ), longitudinal (V_l), shear (V_s), and average sound velocity (V_m) with increasing Cr³⁺ ion concentration

Concentration (x) of Cr ion	V_l (m/s)	V_s (m/s)	V_m (m/s)	ρ (kg/m ³)
0.2	6405.768	4162.62	1168.985	4349.018
0.4	6639.212	4325.87	1318.206	4466.313
0.7	10441.945	4444.73	1278.671	4397.435
0.9	9441.012	4995.42	1405.765	4485.510
1	8871.003	5748.98	1850.092	4289.476

$$\alpha \left(\frac{\text{dB}}{\text{mm}} \right) = 20 \log \left(\frac{S_1}{S_2} \right) \frac{1}{2d}, \tag{15}$$

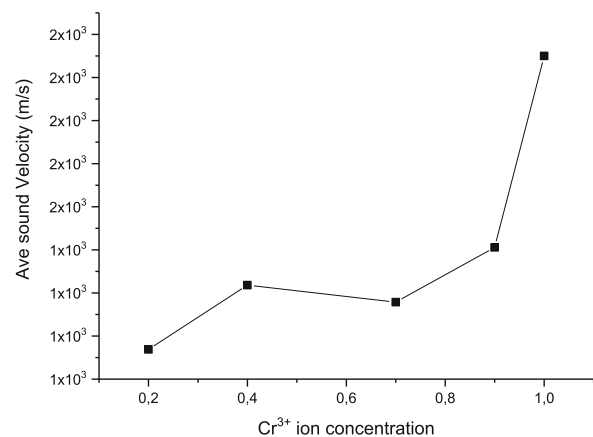
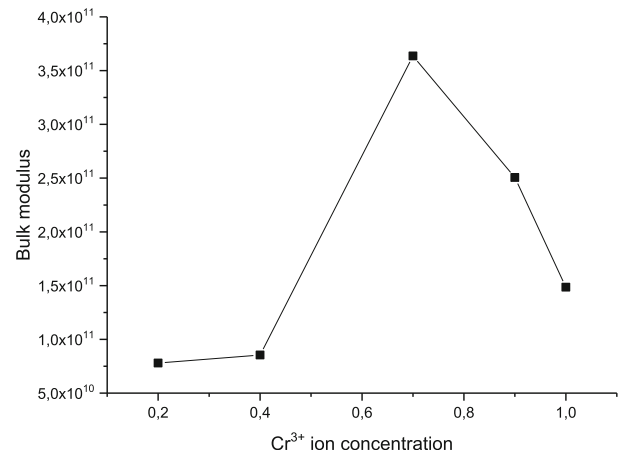
Where S_1 and S_2 are the amplitude of two consecutive back wall echoes, and d is the thickness of the specimen in millimeter. It follows that the velocity of the ultrasound waves in polycrystalline or bulk materials is primarily controlled by elastic modulus and density which is directly related to its microstructure.

The isotropic compressibility changes accordingly, as shown in Fig. 4. The figure shows that the concentration at $x = 0.7$ has a minimum isotropic compressibility and maximum acoustic impedance. The isotropic compressibility is related to the density and longitudinal velocity by the formula $K_s = \frac{1}{\rho V_l}$, and the acoustic impedance of the sound energy is calculated from $Z = \rho V_l$. The isotropic compressibility can be understood by looking at the change in the mean atomic weight due to ion replacement shown in Fig. 5. The continuous decrease in the mean atomic weight with increasing Cr^{3+} ion is due to the difference in ionic weight of Cr^{3+} (51.996) instead of iron (55.847). The variation of the bulk modulus and the average sound velocity with Cr ion concentration is shown in the Appendix, and the two graphs show a behavioral change at $x = 0.7$ in accordance with previous data. In general, a microstructure that strains the lattice or interrupts its continuity reduces the elastic modulus and the velocity of ultrasonic waves. Ferrite materials are made of grain and grain boundaries, and hence, its response to acoustic pulse depends on its microstructure performance. Considering the magnetic nature of ferrite, then magnetocrystalline anisotropy comes into play. The magnetocrystalline anisotropy is a measure for the magnetic energy resistance to the movement of the domain walls, while the domain walls are free to move at a temperature where the magnetic anisotropy is equal to zero. That means the substance undergoes a maximum strain for given stress, or in other words, the longitudinal and shear sound velocity show a decrease with increasing temperature. In the case of an externally applied magnetic field that saturates the sample, the longitudinal and shear sound velocity are slightly higher than the demagnetized state. However, in a mono-domain state, the sound velocities decrease constantly with the increase in temperature (Mazen & Elmosalami, 2011; Bartůněk et al., 2018; Sattar & Rahman, 2003; Muralidhar et al., 1992; Srinivas Rao et al., 2002; Ravinder & Reddy, 2003).

Conclusion

We have characterized the elastic properties of the spinel structure Mn-Cr ferrite with different Cr ion concentration at a fixed sound frequency using the pulse transmission technique. The longitudinal sound velocity showed an increase with increasing Cr ion concentration; the rigidity constant as well as the Debye temperature, the isotropic compressibility, and the acoustic impedance showed behavioral change at Cr ion concentration $x = 0.7$ which is correlated to the intra-ionic distance change due to the replacement of Fe ions with smaller size Cr ions.

Appendix



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Authors' contributions

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Ethics approval and consent to participate

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Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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