Raman Spectroscopic and Electrochemical Measurements of Dynamic Shocked MnFe2O4 Nano-Crystalline Materials

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Research Article

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Abstract

Divalent ferrite nano-crystalline materials subjected to shock wave recovery analysis have received non-debatable attention among the materials scientists due to their outstanding contribution that has fueled the enrichment of high-pressure ferrites science which act as the focal-point of the current research theme. On those lines, we intend to provide the Raman spectroscopic and electrochemical measurements of manganese ferrite nanoparticles (MnFe$_2$O$_4$ NPs) at dynamic shocked conditions. Raman spectroscopic analysis has revealed the stability of the tetrahedral (A-site) and octahedral (B-sites) sites of Mn$^{2+}$ ions as well as Fe$^{3+}$ ions at shocked conditions. Cyclic-voltammetry (CV-analysis) and impedance spectroscopic measurements of shocked samples have demonstrated the electro-chemical properties of MnFe$_2$O$_4$ NPs whose electrical conductivity is found to be reduced while increasing the number of shock pulses. The consolidated results provide the solid evidence that the test sample's crystal structure does not undergo any crystallographic phase transitions or mixed spinel states at shocked conditions.

Introduction

The investigation of knock-on effects influenced by shock waves on materials could make inroads wherein the emerging advancements are looming larger with ever increasing horizons of cutting edge technology, so much so that impending rapid progress is on the cards. On those lines, comprehending the crystallographic and magnetic phase stability of the ferrite nano-materials at high-pressure and high-temperature environments is one of the important hotspot research topics to draw the convincing results of the structural stability of the materials as well as to find new materials of crystallographic configuration with novel functional properties. According to the literature, the majority of the divalent ferrite NPs such as CuFe$_2$O$_4$, CoFe$_2$O$_4$, NiFe$_2$O$_4$, ZnFe$_2$O$_4$, MnFe$_2$O$_4$, MgFe$_2$O$_4$ and BaFe$_2$O$_4$ have been analyzed by X-ray diffraction and Raman spectroscopic techniques at high-pressure conditions and several interesting results have been accounted so far [1–10]. On the one hand, the crystallographic features due to the effect of pressure compression on ferrite NPs have been quite well known while on the other hand, the effect of pressure compression impacted on the ferrite materials’ functional properties such as optical transmittance, dielectric constant, capacitance and electrical conductivity are relatively unknown as compared to the crystallographic details. The remarkable response of nanoparticles on their physical and chemical properties to the external stimuli of high pressure, temperature, gamma rays, and shock waves is quite well known [11–15].

As an integral part of the high-pressure research in the global scenario, dynamic shock waves also have a prominent role in the research of materials science which can create a progressive way forward to understand the stability of materials and provide the scope of tuning the properties of materials including the crystallographic structures which have been well documented to a certain extend in the last couple of decades. Moreover, the materials investigated are both bulk and nano-sized which really emphasize the intuitive ideas such that these materials could be exploited for multiple of applications [16–20]. On the
one hand, in the case of metal oxide nano materials, several interesting crystallographic and magnetic phase transitions have been observed in a variety of materials such as TiO$_2$, ZrO$_2$ and Co$_3$O$_4$[21–23]. On the other hand, in the case of divalent ferrite NPs such as ZnFe$_2$O$_4$, NiFe$_2$O$_4$ and MnFe$_2$O$_4$, stable crystallographic and magnetic phases have been witnessed at shocked conditions which have also thrown a few more fascinating results [24–26]. Interestingly, ZnFe$_2$O$_4$ NPs exhibit paramagnetic to superparamagnetic switchable magnetic phase transitions in accordance with the counts of shock pulses of 0, 50,100 and 150 [24]. NiFe$_2$O$_4$ and MnFe$_2$O$_4$ NPs have stable crystallographic and magnetic states at shocked conditions [25, 26]. But there is no report available so far on Raman spectral and electrochemical properties for NiFe$_2$O$_4$ and MnFe$_2$O$_4$ NPs at shocked conditions. In order to have access to the fabrication of electronic devices, knowing the electronic transport properties and their stabilities of materials are highly required since there are lots of changes that can occur in the transport properties as and when sudden environmental changes happen [27, 28]. Moreover, at shocked conditions, there have been remarkable changes observed for a few structurally stable materials [29, 30]. Hence, we intend to investigate the electrical transport properties of technologically important MnFe$_2$O$_4$ NPs at shocked conditions which could make a probable justification on the stability of crystal structure and the associated material properties contributing to their performances at shocked conditions. Hence, herein as the continuation of our previous report, we account the Raman spectroscopic and electrical transport properties of MnFe$_2$O$_4$ NPs at shocked conditions. The title material deserves several technologically important applications such as site-specific drug delivery, magnetic-storage devices, sensors, energy storage applications, magnetic resonance imaging and photo-magnetic materials, RADAR and high-speed data transfer communication systems [31–33]. MnFe$_2$O$_4$ NPs crystallize with the cubic normal spinel space group at ambient temperature (Mn$^{2+}$ occurred only in tetrahedral site (A-site) of MnFe$_2$O$_4$) and also it can crystallize with the inverse spinel as well as mixed spinel cubic phase while changing the synthesis process (For inverse spinel, Mn$^{2+}$ occurred only in octahedral site (B-site) of MnFe$_2$O$_4$) and in the case of mixed spinel, Mn$^{2+}$ ions are present both in the tetrahedral and octahedral sites [32, 33]. Interestingly, the transition from the normal spinel to the mixed spinel of ZnFe$_2$O$_4$ NPs has been observed at shock wave loaded conditions and the corresponding Raman spectroscopic results have been communicated elsewhere. Hematite (Fe$_2$O$_3$ NPs) also show significant enhancement of electrical conductivity at shocked conditions while being in the same crystallographic phase [35, 36]. In the case of MnFe$_2$O$_4$, possibilities of such kind of changes are on the higher scale at shocked conditions since both the ferrites belong to the ZnFe$_2$O$_4$ type normal spinel category at room temperature. Herein, we account the details of the Raman and electrochemical properties of the control and shocked MnFe$_2$O$_4$ NPs.

**Experimental Section**

The synthesis process of MnFe$_2$O$_4$ NPs and the procedure of shock wave loading have been presented in the previous paper and provided as a supplementary data in supplementary section [26]. The electrochemical measurements have been carried out by utilizing an electrochemical workstation (SP-150, BioLogic Science Instruments, France) at ambient temperature. In order to perform electrochemical
analysis, the required electrode has been made by blending 80 wt% of the obtained active material (MnFe$_2$O$_4$), 10 wt % of the conductive carbon black and 10 wt% of polyvinylidene fluoride (PVDF) binder along with the solution of 0.3 mL N-methylproline (NMP). The obtained uniform slurry has been painted on the well-cleaned nickel foam (1 cm × 1 cm) which has been kept to air dry for 24 hours at ambient conditions. For the preparation of the electrode, the required mass of the active material (1 mg) has been measured by a digital weighing device. The electrochemical measurements have been performed making use of a three-electrode cell possessing 3 mol electrolyte solution of KOH such that the combination of these have been made use of as the working electrodes wherein Pt wire and Ag/AgCl in the saturated KCl have been chosen as the counter and as the reference electrode, respectively. Shock wave loaded sample electrodes of counts 50, 100 and 150 have been prepared. The cyclic analysis of voltammetry (CV) has been performed for various applied potential ranging from 0 V to 0.5 V. Followed by the cyclic voltammetry, electrochemical impedance spectroscopic (EIS) analysis has been accomplished for the frequency ranging between 0.01 Hz and 1 kHz while the amplitude of 5 mV has been maintained as the open circuit potential.

**Results And Discussion**

**Raman Spectroscopy**

Raman spectroscopy is one of the prime tools being used to investigate the crystallographic phase details, lattice disorder, charge and spin-lattice coupling and local cation distributions [32]. Especially in ferrite materials, the transport properties are partially dependent on the cation distributions in the tetrahedral (A-site) and octahedral (B-site) sites. Hence, the Raman spectroscopic analysis (LabRAM HR Evolution, France) has been carried out for the samples of both the control and shocked. The obtained profiles of the Raman spectra are presented in Fig. 1.

According to the factor group analysis, cubic MnFe$_2$O$_4$ spinel has five Raman active modes as that of A$_{1g}$, E$_g$ and 3F$_{2g}$. Among the Raman active modes, A$_{1g}$ and 3F$_{2g}$ Raman modes belong to the tetrahedral (A-site) and E$_g$ Raman mode belongs to the octahedral (B-site). As seen in Fig. 1, the control sample has four Raman modes such as A$_{1g}$, F$_{2g}$ (2), E$_g$ and F2g (1) whose corresponding Raman positions are 650, 407, 293, and 221 cm$^{-1}$, respectively and the observed Raman bands are well-matched with the previous reports of the normal spinel MnFe$_2$O$_4$ NPs [32]. No considerable change is obtained under shock loading which is clearly evident for the mere naked eye.

But, while seeing the zoomed versions of the tetrahedral and the octahedral sites, slight changes are observed in terms of the ratio of Raman intensity that is between the tetrahedral and the octahedral sites in accordance with the number of shock pulses. As reflected in Fig. 2, the peak of F$_{2g}$ (1) for the control MnFe$_2$O$_4$NPs has maximum intensity than that of the other Raman peaks such as E$_g$ and F$_{2g}$ (2) which remain the same for all the other shocked conditions. But, E$_g$ Raman band intensity increases slightly on increasing the number of shock pulses whereas F$_{2g}$ (1) Raman intensity reduces on increasing the
number of shock pulses. Moreover, the above-mentioned Raman bands have undergone a shift towards the lower frequency corresponding to the counts of shock pulses and the respective Raman shifts are depicted in Fig. 3.

The observed changes both in Raman band intensity and Raman shifts in accordance with the number of shock counts could have lead to lattice defects along with the redistribution of divalent and trivalent cations in the A and B sites wherein the interaction between oxygen and cations in the octahedral as well as the tetrahedral positions are strongly affected [36]. In the normal spinel structure, the divalent ions (Mn$^{2+}$ in MnFe$_2$O$_4$) occupy the A site only whereas the trivalent ions (Fe$^{3+}$ in MnFe$_2$O$_4$) occupy the B sites only. Considering the structure of inverse spinel, half of the B sites are occupied by the divalent ions while the rest of the B sites are occupied by the trivalent ions in addition to the occupancy of the entire A sites [32]. At high-pressure and high-temperature conditions, the migration of Mn$^{2+}$ ions from the A to the B sites can occur. Theoretically, this would mean an effective increase in unit cell volume with which a significant XRD peak shift would have occurred for the higher inversion degree for MnFe$_2$O$_4$ [37]. Even a minor number of Mn$^{2+}$ changes to Mn$^{3+}$ ions in the B-sites, it significantly affects the XRD peak positions [26]. But, such changes have not been observed in the XRD peak positions at shocked conditions. Hence, it is confirmed that there is no inverse spinel structure that has been formed at shocked conditions. Therefore, the observed changes in the Raman band intensity may be due to the local lattice distortions. But, the cubic normal spinal phase of MnFe$_2$O$_4$ remains the same even after 150 shocked conditions which are well corroborated with the previously reported XRD results [26]. It could be noted that, at shocked conditions, the technologically important materials such as TiO$_2$ and Co$_3$O$_4$ NPs undergo significant Raman shifts due to the occurrence of the crystallographic phase transitions [21, 23].

Cyclic-voltametry Analysis

The Cyclic-voltammetric study has been performed to analyze the specific capacitance of the title material at shocked conditions which are presented in Fig. 4. The variation of the electrochemical performance of the control and shocked samples have been analyzed with different scan rates such as 5, 10, 20, 50, and 100 mV s$^{-1}$ in the potential range of 0 - 1.0 V. As seen in Fig. 4, while increasing the scan rate, the positive shifts are observed in the redox peaks with respect to the anodic and cathodic potentials as well as the increased loop area with respect to the scan rate which is the usual behavior found in materials of ideal capacitive behavior [38].

It could be noted that the CV loop shapes and the loop widths of the control and shocked samples are almost similar while at different rates of shocked conditions. From an initial viewpoint, it could be seen that there is no significant change that has been observed in the capacitance value at shocked conditions. Furthermore, we have calculated the specific capacitance of both the control and shocked MnFe$_2$O$_4$ NPs using the standard formulation and the obtained values of specific capacitance in accordance with the scan rates are portrayed in Fig. 5. As seen in Fig. 5, the values of specific capacitance are reduced while increasing the scan rate for all the samples. Even at a lower scan rate, the
electrolyte ions have adequate time to penetrate the pores of the electrode material because of their higher value of specific capacitance.

For better understanding the shapes of CV loops and the values of specific capacitance with respect to the number of shock pulses, the comparison loops are presented in Fig. 6a. All the loops are similar in their shape for the samples such that no change is observed in their respective values of potential with respect to the number of shock pulses and the corresponding values of specific capacitance with respect to the number of shock pulses are plotted as presented in Fig. 6b. Hence, it is clear that no cationic redistributions appear at shocked conditions and the observed slight changes may be due to the lattice defects and surface changes of the particles. It could be noted that the observed values of specific capacitance with respect to the number of shock pulses have not changed significantly.

**Electrochemical Impedance Spectroscopic Analysis**

Furthermore, we have performed the electrochemical impedance spectroscopic analysis to understand the behavior of electrical resistance under shock loading. Note that the variation of the microstructure, lattice disorder and cation redistribution can influence changes in the electronic transport properties in such a way that several high-pressure compression experiments have been performed on this theme providing several interesting results [11]. Hence, electrochemical impedance spectroscopic analysis (EIS) has been performed and the obtained Cole-Cole plots of the control and shocked samples are portrayed in Fig. 7. As per the normal spinel crystal structure of MnFe$_2$O$_4$ NPs, the interactions of the Mg$^{2+}$ ion in the tetrahedral sites and Fe$^{3+}$ ions in the octahedral sites are quite low hence it has lower electrical conductivity. As seen in Fig. 8, the control sample gives rise to an incomplete semi-circular arc and shocked samples also show a similar trend of semi-circular arcs with slight changes. Especially in the grain resistance portion (higher frequency region), the width of the first semi-circular arc is increased with respect to the number of shock pulses and the observed zoomed version of the Cole-Cole plots are depicted in Fig. 8. As the semi-circular arc width increases, the electrical resistance also increases. Hence, it is clear that the impact of applied shock waves has increased the values of electrical resistance (bulk grain resistance) with respect to the number of shock pulses which may be due to the formation of lattice defects in the cubic spinel, grain boundary region that has been disordered resulting in a tremendous increase in electron scattering [35, 38]. The mobility of charge carriers have a high dependency on temperature and pressure such that the reduction in mobility could have enforced a reduction in the electrical conductivity due to the change occurring in activation energy. Similar results of slight changes in the values of saturation magnetization have been witnessed in accordance with the counts of shock pulses [26].

**Conclusion**

Drawing the summary of the present experimental results, Raman spectroscopic and electro-chemical properties have been investigated for the control and shocked samples. Raman spectroscopic results
provide a clear justification on the crystallographic phase stability of the cubic normal spinel lattice of MnFe$_2$O$_4$ NPs at shocked conditions which is well corroborated with the previously reported X-ray diffraction studies. Electrochemical measurements such as cyclic-voltammetry and impedance spectroscopic measurements have provided the possible authentication on the stability of the electrochemical properties of the title material at shocked conditions so that MnFe$_2$O$_4$ NPs are proposed as a good fit for electronic device fabrication.

**Declarations**

**Conflicts of interest**

The authors declare that they have no conflict of interest.

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Figures

![Figure 1](image-url)
Raman spectra of the control and shocked MnFe2O4 NPs

Figure 2

Zoomed version of the control and shocked MnFe2O4 NPs
Figure 3

Raman shifts of F2g (1), Eg and F2g (2) with respect to the number of shock pulses
Figure 4

CV curves of the control and shocked MnFe2O4 NPs
Figure 5

Plots of specific capacitance of the control and shocked MnFe2O4 NPs with respect to the scan rates.
Figure 6

(a) Comparison of CV curves of the control and shocked samples at 100 mVs-1 (b) The values of specific capacitance with respect to the number of shock pulses

Figure 7

Cole – Cole plots of the control and shocked MnFe2O4 NPs
Figure 8

Zoomed version Cole – Cole plots of the control and shocked MnFe2O4 NPs