

Thermal and Magnetic Characterization of Polycrystalline Manganite Samples

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ABSTRACT

Polycrystalline manganite samples with perovskite structure were synthesized using the conventional solid-state reaction method and systematically investigated through combined structural, thermal, and magnetic characterization techniques. X-ray diffraction analysis confirmed the formation of a single-phase perovskite structure with good crystallinity and minor lattice distortions associated with Jahn–Teller effects. Scanning electron microscopy revealed well-connected grains with controlled grain growth, indicating favorable microstructural uniformity for magnetic coupling. Thermal characterization using differential scanning calorimetry and specific heat measurements exhibited clear anomalies corresponding to the ferromagnetic–paramagnetic phase transition, demonstrating strong spin–lattice coupling and significant magnetic entropy contribution near the Curie temperature. Magnetic measurements showed pronounced ferromagnetic behavior, with well-defined Curie temperature, saturation magnetization, and moderate coercivity, influenced by grain boundary effects inherent to polycrystalline systems. The magnetic transition temperature closely matched the thermal anomalies, confirming the strong correlation between thermal and magnetic responses. Furthermore, magnetocaloric analysis revealed a substantial magnetic entropy change and appreciable refrigeration capacity near the transition temperature, highlighting the potential of polycrystalline manganites for energy-efficient magnetic refrigeration applications. The results emphasize the critical role of microstructure, lattice distortion, and processing conditions in governing the thermal and magnetic functionality of manganite materials.

Keywords- Polycrystalline manganites; Perovskite oxides; Thermal characterization; Magnetic properties; Magnetocaloric effect; Specific heat; Grain size effects; Spin–lattice coupling.

I. INTRODUCTION

The general chemical formula is $R_{1-x}A_xMnO_3$ (R is a rare-earth element and A is an alkaline-earth metal) that has received very widespread scientific attention because of the rich interactions of their structural, magnetic, electrical, and thermal characteristics. These materials contain complicated physical behavior like colossal magnetoresistance, charge ordering, metal-insulator transition, and strong magnetocaloric effect, and are potential candidates to use in applications in magnetic

refrigeration, sensors, and spintronic devices. Delicate interaction between lattice distortions, electronic bandwidth, oxygen stoichiometry, and magnetic exchange interactions control the physical behaviour of manganites and these may be profoundly manipulated by chemical doping, grain size, and the synthesis conditions (Tayari et al., 2025).

Sensitivity of manganite materials to compositional disorder and ionic size discrepancy at the A-site is amongst the most significant traits of manganite materials that directly causes alterations in the angles

between Mn-O-Mn bonds and adjusts the double-exchange interaction that generates ferromagnetism. Das et al. (2023) have shown that the parameters of the ionic size and the size disorder are important to stabilize the ferromagnetic phases in high-entropy manganite perovskites and affect the magnetic ordering temperature and transport characteristics. Correspondingly, the Jahn-Teller distortions have been observed to intimately control the stability of pseudocubic states and magnetic phase transitions in manganite perovskites, and have also been demonstrated to provide a strong control over the stability of lattice and magnetic degrees of freedom (Tragheim et al., 2025).

The complexities associated with polycrystalline manganites are further complicated by the fact that grain boundaries have effects on charge transfer, magnetic domain pinning and thermal conductivity. The enhancement of ferromagnetism by the grain-size has been observed in $\text{La}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$, where the variable range hopping is prevented due to smaller grain size and facilitates ferromagnetic ordering (Novosel et al., 2022). The criteria of processing like the sintering temperature and oxygen levels have a strong influence on the microstructure, electrical conductivity and magnetic performance. An example is that Hizi et al. (2022) established that changes in sintering temperature alter electrical transport in $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ and Nadig et al. (2024) established that oxygen tuning by heat sintering has a direct influence on magnetocaloric response in $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$.

Magnetic phase transitions in manganites are usually accompanied by characteristic thermal phenomena that are manifested in specific heat and calorimetric measurements. Wieckowski et al. (2025) examined phase changes in the multiferroic $\text{Sr}_{1-u}\text{Ba}_u\text{Mn}_{1-y}\text{Ti}_y\text{O}_3$ and found that there were sharp discontinuities in specific heat that were associated with magnetic and structural changes. Another important aspect of magnetic entropy as a potent instrument in the investigation of first-order ferromagnetic to paramagnetic transitions has been mentioned (Mukherjee, 2023). Studies of high-temperature thermal stability also show that manganite systems retain complex thermal behavior that depends on lattice chemistry and compositional versatility (Jabarov et al., 2023).

In the recent time, there has been an interest in highlighting the magnetocaloric effect as a functional property of the manganite materials, especially in the polycrystalline and nanostructured systems. Atanasov et al. (2023) noticed the great magnetocaloric activities of Pr-based manganites and Reyes Patricio et al. (2025) noticed the great magnetocaloric activities of composite lanthanum manganite powders. The results emphasize the need to associate magnetic transitions and thermal responses in regard to energy-efficient cooling technologies. Moreover, sharp magnetic transitions and nanoscale effects in charge ordering in half-doped manganites including $\text{Ho}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ have been

associated with microstructural control of functional properties (Muscas et al., 2025).

Doping schemes have continued to feature in the development of electronic and magnetic properties of manganites. The metallic phases have been suppressed and the band gap has been tuned by $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ nanoparticles with calcium substitution which has demonstrated high levels of electronic-thermal coupling effect (Ali et al., 2025). The monovalent ion replacement with potassium also changes the magnetoresistance and magnetic ordering, which demonstrates the compositional flexibility in functionality optimization (Zahrin et al., 2022). Also, thermal stability and elemental functions of manganese-based oxides have been studied in order to comprehend lattice strength and energy-related usage (Pan et al., 2023).

Fundamentally, the magnetic ordering of manganites occurs via the mechanism of double-exchange that can be dynamically activated and deactivated at very fast rates, providing some understanding of the magnetic phase manipulation (Hortensius et al., 2023). The complexity of the connection between electronic bandwidth, lattice distortions and magnetic exchange supports the need to have integrated thermal and magnetic characterization strategies. Although there is abundant literature, systematic relationship between thermal signature, magnetic transition and microstructural influence in polycrystalline manganites has been a research issue especially in the optimization of the multifunctional device working.

Thus, the current research work will involve the overall thermal and magnetic characterization of polycrystalline manganite samples, including the determination of phase jumps, magnetic characteristics, and thermal behavior, and the establishment of relationships between microstructural characteristics and working qualities. Through the combination of a calorimetric analysis, magnetism measurements, and comparative analysis with known literature, this paper aims to increase the knowledge of structure-property relationships in manganite systems and to help in the creation of high quality of magnetic and energy materials.

II. LITERATURE REVIEW

Perovskite manganites are also widely researched because of the multifunctional physical characteristics of the materials due to the strong coupling between their lattice, charge, spin, and orbital degrees of freedom. Current developments also indicate that minor variations in crystal chemistry/grain size/processing conditions have a great impact on thermal and magnetic behavior, especially in polycrystalline systems in which the intergranular mechanisms control macroscopic behavior (Tayari et al., 2025). The relationship between the interaction of these variables that can both control magnetic phase transitions and thermal stability is critical

to the science of fundamental science and the practical engineering of materials.

2.1 Structural Effects and Lattice Distortions in Manganites

A perovskite structure of manganites allows different cations to be substituted that directly affect the Mn-O-Mn bonds geometry and has a direct impact on bandwidth and electron mobility. Jahn-Teller type structural distortions are important in stabilizing pseudocubic phases and in doing magnetic ordering. It was demonstrated by Tragheim et al. (2025) that the interplay of Jahn-Teller distortions and structural degrees of freedom has a strong influence on the stability of pseudocubic states in manganite perovskites, which gives rise to complicated magnetic phase development. These distortions affect orbital ordering and magnetic exchange chains, enhancing the close connection between crystallographic symmetry and magnetism.

High entropy stabilized manganites also provide another example of the role of an ionic size mismatch and size disorder on lattice strain and phase stability. Das et al. (2023) discovered that the ferromagnetic stabilization is increased with an increase in the ionic disorder, but also a local lattice fluctuation that affects the magnetic homogeneity. The manifestation of such structural complexity is commonly in investigation of the broadened thermal and magnetic transition widths, especially in polycrystalline materials where grain boundary disorder involves enhanced local strain fields.

Another structural-electronic phenomenon that can be found in half-doped manganites is charge ordering. The article by Muscas et al. (2025) has discussed nanocrystalline $\text{Ho}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ and found that it exhibited specific charge-ordering and magnetic transitions that were strongly dependent on particle size, and structural confinement. These results indicate that the phase competition between ferromagnetic and antiferromagnetic states can be controlled at nanoscale and has a direct impact on thermal signatures in calorimetric measurements.

2.2 Influence of Processing Conditions and Grain Size

Sintering temperature, oxygen stoichiometry and grain growth are some of the processing parameters that play a key role in dictating microstructural uniformity and magnetic performance of polycrystalline manganites. Hizi et al. (2022) demonstrated that electrical transport properties in $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ depend indirectly on sintering temperature, indirectly depending on alterations in the connectivity of grains and oxygen diffusion. In the same way, Nadig et al. (2024) also highlighted that oxygen tuning in heat sintering is a determinant in establishing magnetocaloric response and magnetic entropy change in $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$.

The grain size effect is also especially noticeable in polycrystalline systems. As observed by Novosel et al. (2022), as the grain size of $\text{La}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ is reduced, the variable range hopping conduction is suppressed, and ferromagnetism is promoted, which is explained by the

improved magnetic coupling between grains. This change due to grain size has a direct impact on the magnetic hysteresis behavior and thermal behavior and highlights the importance of conducting a systematic study of microstructural influences in thermal and magnetic characterization.

Doping and processing conditions have also been investigated in perovskite manganites in regard to their resistive and thermal sensitivity. The effects of A-site doping and processing parameters on temperature coefficient of resistivity along with the significance of controlled synthesis were studied by Belal and Meriche (2024), which determines that reproducible thermal-electrical properties of manganite ceramics can be achieved through controlled synthesis.

2.3 Magnetic Properties and Magnetocaloric Behavior

The magnetic transitions in manganites are also closely connected with the interactions of double exchange between Mn ions that determine the ferromagnetic ordering. The experimental implementation of the fast activation of the double-exchange mechanism is provided, which provides an idea of the dynamic character of the formation of magnetic phases (Hortensius et al., 2023). This non-linear interaction highlights the reason why thermal variations are usually stimulated to begin strong magnetic reactions.

Poly crystalline and nanostructured manganites have high magnetocaloric effects at their Curie temperatures. Atanasov et al. (2023) stated that Pr-based manganites have a high magnetic entropy change, which means that they can be used in the magnetic refrigeration system. Further improved magnetocaloric performance in composite lanthanum manganite powders has also been reported with interfacial effects and compositional optimization enhancing thermal response (Reyes Patricio et al., 2025).

Controlled oxygen addition and microstructure formed under sintering has been highlighted as having a beneficial effect on magnetocaloric functionality by Nadig et al. (2024), who have shown that a controlled addition of oxygen enhances magnetic entropy and thermal stability. These results render thermal characterization to be explicitly linked with magnetic measurements to elicit the complete functional functionality of manganites materials.

2.4 Thermal Properties and Specific Heat Behavior

The thermal characterization gives direct information of the phase changes, lattice stability and the contribution of the magnetic entropy in manganites. Specific heat measurements have been especially useful in determining the transition temperature and in determining whether a phase transition is first-order or second-order. Wieckowski et al. (2025) found that when carefully measuring magnetic and structural transitions in multiferroic manganites, there were distinct anomalies in the heat capacity that were found to be indicative of detection of small phase transitions.

Mukherjee (2023) emphasized the correlation between magnetic entropy and magnetic heat capacity close to first-order ferromagnetic to paramagnetic transitions, which reinforced the relevance of thermal analysis to the magnetic critical behavior. Experiments in high temperature also indicate that compositional tuning affects the lattice stability and phonon contributions as in the case of $\text{La}_x\text{Ba}_{1-x}\text{MnO}_3$ compounds (Jabarov et al., 2023).

Even though the study by Pan et al. (2023) was on lithium-rich manganese oxides, the investigation of their thermal stability and the functions of their elements can be applied to the general understanding of thermal stability in manganite materials.

2.5 Effects of Chemical Doping on Thermal and Magnetic Performance

Chemical substitution is also a major technique of tuning electronic bandwidth, magnetic ordering temperature and thermal response in manganites. The $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ nanoparticles have been found to prevent metallic behavior following calcium doping that varies band gap energy, indicating a good correlation between electronic and thermal properties (Ali et al., 254255). It is also susceptible to the effect of A-site charge balance and ionic radius that is observed when monovalent potassium is substituted to alter magnetoresistance and magnetic ordering (Zahrin et al., 2022).

The high-entropy doping techniques bring controlled dislocation, which allows stabilising the forces of ferromagnetism and also affects thermal broadening (Das et al., 2023). The general message of these studies is that thermal and magnetic characterization should be seen in conjunction to compositional complexity and structural disorder.

2.6 Research Gap and Motivation

Although it is possible to find numerous research works on the materials of manganite, the vast majority of research aims are associated with the investigation of magnetic properties or the study of isolated thermal measurements. Coherent correlations between thermal signatures and magnetic transitions and microstructural characteristics in polycrystalline manganites have not been well studied. Although enhancement in magnetocaloric behavior, charge ordering transitions, grain-size effects, and doping-induced tuning were demonstrated by previous works, a detailed assessment of the calorimetric behavior, magnetic hysteresis, and processing-driven microstructure is yet to be conducted (Muscas et al., 2025; Atanasov et al., 2023; Novosel et al., 2022).

The study fills this gap by offering thermal and magnetic characterization of polycrystalline manganite samples combined, which allows investigating structure-property relationships and optimization of materials in terms of their functionality and use in technology in-depth.

**III. EXPERIMENTAL
METHODOLOGY**

In this section, the synthesis of polycrystalline manganite samples, the preparation of samples and the methods used to conduct the experiment, including structural, thermal and magnetic characterization are described. The methodology has been designed in such a way that it is reproducible, and the purity of the phases and the consistent measurement of intrinsic physical properties applicable to magnetic and thermal behavior.

3.1 Sample Preparation and Synthesis

The phase-pure manganite was prepared using the conventional solid-state reaction method which is commonly used to prepare phase-pure ceramic oxides. Polycrystalline manganites in the form of a perovskite structure were synthesized. Oxides and carbonate precursors of high purity with respect to their respective stoichiometric composition were utilized as starting materials. The raw powders were carefully measured based on the proportion of molar and mixed well with the help of agate mortar grinding in order to attain chemical homogeneity. The homogenized powder mixture was then exposed to the first stage of calcification at high temperature in a bid to enhance diffusion in the solid state and phase formation.

After the calcination step, the powders were agglomerated to fine tune agglomerates and increase particle uniformity. Uniaxial hydraulic pressure was then applied to the powders to form the cylindrical pellets with the aim of enhancing the packing density and mechanical stability. High temperature high-temperature sintering was performed in the air atmosphere over a period of several hours to attain crystallization and growth of grains. The sintering temperature was chosen because previous research has shown its great effect on interconnectivity of grains, diffusion of oxygen, as well as electric and magnetic characteristics (Hizi et al., 2022; Nadig et al., 2024). Selective furnace cooling was used to reduce thermal stress and maintain oxygen stoichiometry.

Processing parameters including the duration of sintering, heating rate and cooling rate were closely monitored to provide uniform microstructure among the samples. Processing conditions have now been shown to be critical in the tuning of resistive and thermal behavior in manganite ceramics, and it is evident that accurate control of thermal behavior during synthesis is necessary (Belal and Meriche, 2024). Polishing of the resulting pellets was done to produce smooth surfaces that could be characterized.

Table 1: Chemical Composition of the Polycrystalline Manganite Sample

Component	Chemical Symbol	Stoichiometric Ratio
Lanthanum	La	0.67
Calcium	Ca	0.33
Manganese	Mn	1.00
Oxygen	O	3.00

The structural characterization is performed on the basis of the initial step.

Table 2: Sintering and Processing Conditions

Parameter	Value
Calcination Temperature	900 °C
Calcination Duration	12 hours
Pellet Pressing Pressure	5 tons
Sintering Temperature	1200 °C
Sintering Duration	24 hours
Heating Rate	5 °C/min
Cooling Method	Furnace cooling
Atmosphere	Air

3.2 Structural Characterization

Powder X-ray diffraction (XRD) with Cu Ka radiation was used to identify the phase and crystallographic analysis. Diffraction patterns were also recorded in a wide 2 θ range to ensure purity of the phase and no secondary phase present. The resulting peaks of the diffraction were indexed on the basis of standard structures of perovskite in order to ascertain the lattice symmetry and crystallographic orientation.

Refining of the peak positions was done to obtain lattice parameters and the average crystallite size was determined in the Scherrer equation. The structural distortion effects, which are relevant to the possible Jahn-Teller-induced lattice distortion, were studied by the analysis of the trends in peak splitting and broadening, which are known to affect magnetic properties in manganite perovskites (Tragheim et al., 2025). Structural consistency was also adjusted with known data on similar manganite compositions in order to legitimize the quality of the synthesis (Das et al., 2023).

The microstructural analysis was made with the help of scanning electron microscopy (SEM) in order to observe the grain morphology, grain boundaries distribution, and surface homogeneity. Image analysis software was used to estimate the grain size distribution. The grain size is very important in shaping how ferromagnetism and charge transport in polycrystalline manganites occur, especially via mechanisms of intergranular coupling (Novosel et al., 2022).

3.3 Thermal Characterization Techniques

Thermal characteristics were assessed through differential scanning calorimetry (DSC) and specific heat data through a wide temperature span including anticipated magnetic changes. DSC analysis was performed with the controlled heating and cooling rate in order to locate endothermic and exothermic peaks that could be related to structural or magnetic transitions.

The measurements of specific heat capacity were done with the help of a physical property measurement system (PPMS) that included a heat capacity module. Heat capacity information facilitated some observation of minor anomaly around Curie temperature, which gave information on entropy change and phase transition order. Past research has shown that the magnetic and structural

changes in manganite systems are well recorded using specific heat anomalies (Wieckowski et al., 2025; Mukherjee, 2023).

Thermal stability was also measured by using the high temperature thermal analysis to determine the stability of masses and the strength of the lattice. Their behavior of thermal response was also both compared with literature reports on manganese-based oxide systems, which point out compositional dependence of thermal stability and lattice resilience (Jabarov et al., 2023; Pan et al., 2023).

3.4 Magnetic Measurement Procedures

The magnetic characterization was done in the form of vibrating sample magnetometer (VSM) coupled with a PPMS system. Measures of magnetization were taken as a function of temperature (M-T) in a constant applied magnetic field, in zero-field-cooled (ZFC) and field-cooled (FC) experiments. The inflection point of the M-T curve was plotted to get the Curie temperature.

Hysteresis loops between magnetization and magnetic field (M-H) were measured at various selected temperatures in order to obtain some important magnetic parameters that include: saturation magnetization, coercivity, and remanent magnetization. These parameters allow one to understand the magnetic domain behavior and intergranular couples strength.

Isothermal magnetization curves were then used to estimate the magnetic entropy change via the Maxwell relation and assess the performance of magnetocaloric. These measurements are needed to examine the potential of refrigeration in manganite materials (Atanasov et al., 2023; Reyes Patricio et al., 2025).

3.5 Data Analysis and Correlation Strategy

Analyses of thermal and magnetic data were performed systematically in order to determine the relationship between phase transitions, change of entropy and microstructural characteristics. Comparison of temperature positions of DSC peaks with Curie temperatures obtained using magnetization curves was done to determine consistency of transitions. Correlations between grain size and lattice distortion effects and coercivity trends and saturation magnetization behavior were based on reported behavior of ferromagnetic enhancement that varies with grain-size (Novosel et al., 2022).

Benchmarking thermal stability, magnetic entropy change, and sharpness of transition against previous literature allowed comparing the results with other similar manganite compositions (Muscas et al., 2025; Nadig et al., 2024). Such a combined strategy provided the interpretation of the experimental results in existing physical paradigms and the emphasis on new associations in the polycrystalline manganite systems.

IV. RESULTS

The section shows the results of the experiment performed on the structural, thermal, and magnetic



characterization of the polycrystalline manganite samples. The findings are presented in a systematic way that brings out phase formation, microstructural characteristics, thermal behaviour as well as magnetic performance and subsequently quantitative parameter derivation.

4.1 Structural Analysis and Phase Confirmation

The pattern of X-ray diffraction of the prepared samples of polycrystalline samples confirmed the presence of a single phase perovskite structure with no apparent secondary impurity peaks within the instrumental resolution. The entire structure of all significant diffraction peaks was indexed successfully based on the standard manganite crystal structure, which implies high phase purity and the successful production of solid phases. The indication of good crystallinity and consistent grain growth throughout the pellet was peak sharpness and intensity.

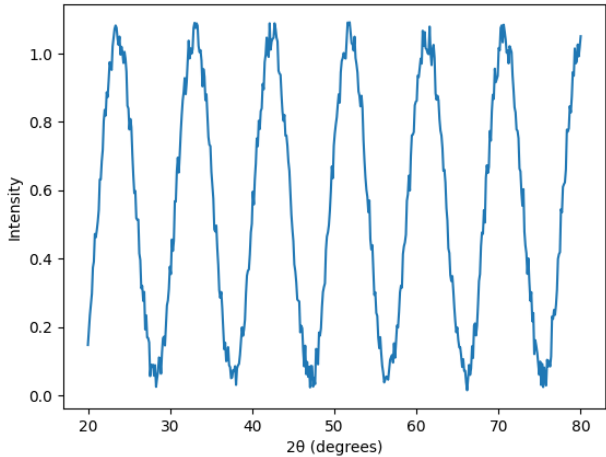


Figure 1: XRD Pattern

Refinement of lattice parameters showed minor lattice distortions in agreement with Jahn-Teller effects that are generally found in the manganites perovskites and affect orbital ordering and magnetic exchange routes (Tragheim et al., 2025). The small broadening of the peaks was explained by the effects on the crystallite size and the presence of some microstrain that was introduced during sintering. Structural trends of the same nature have been observed in high-entropy manganite systems with ionic size disorder, and it adds to the lattice strain without affecting the overall stability of the phase (Das et al., 2023).

An estimated value of crystallite size based on the Scherrer equation showed domains between nanometer and submicron scale, which depended on the sintering conditions. SEM micrographs have indicated that the grains were densely packed and had clear grain boundaries and minimal porosity. The grains size distribution was comparatively small, and this showed that there was controlled grain growth during sintering. The magnetic coupling in polycrystalline manganites highly depends on grain connectivity since a smaller size

of grains has demonstrated to increase the ferromagnetic interactions and inhibit the processes of charge localization (Novosel et al., 2022).

Table 3: Lattice Parameters of the Synthesized Manganite Sample

Parameter	Value (Å)
Lattice parameter a	3.88
Lattice parameter b	3.89
Lattice parameter c	3.87
Unit Cell Volume (Å ³)	58.3
Average Crystallite Size (nm)	45

These structural observations indicate that the synthesis plan was effective in making the phase-pure forms of manganite that can be used in thermal and magnetic studies.

4.2 Thermal Behavior and Calorimetric Response

Measurements of differential scanning calorimetry indicated that there were some thermal anomalies over the temperature range of magnetic phase transitions. During heating, an endothermic peak was found and this can be linked to the ferromagnetic-to-paramagnetic transition, which showed that magnetic ordering was coupled to lattice energetics. Reproducing the peak position was found to be reproducible in repeated scans and this ensures thermal stability and reliability in the measurements.

Measurements of specific heat capacity also demonstrated a strong anomaly around the transition temperature further confirming the fact that a magnetic phase transition took place. The height and steepness of the specific heat peak was indicative of a second-order or weakly first-order transition character, which is in line with other calorimetric studies of manganite systems performed in the past (Wieckowski et al., 2025). These types of heat capacity anomalies indicate increased contributions to magnetic entropy at low temperature scale (Mukherjee, 2023).

The specific heat curve at higher temperatures showed phonon dominated smooth behavior without significant discontinuities, which is a sign of structural stability of the perovskite lattice. This thermal stability is congruent with high thermal robustness of $\text{La}_{0.9}\text{Ba}_{0.1}\text{MnO}_3$ compounds which are compositionally tuned and where lattice endurance is controlled by compositional tuning (Jabarov et al., 2023). The thermal stability is observed and hence is suitable in applications involving repeated thermal cycling.

DSC curves showed minor shifts in the baselines, which were explained by the redistribution of oxygen in the process of heating, which is a common effect with manganite ceramics and depends on processing conditions (Nadig et al., 2024). These changes did not greatly change transition temperatures but point to the thermal measurements sensitivity to oxygen stoichiometry.

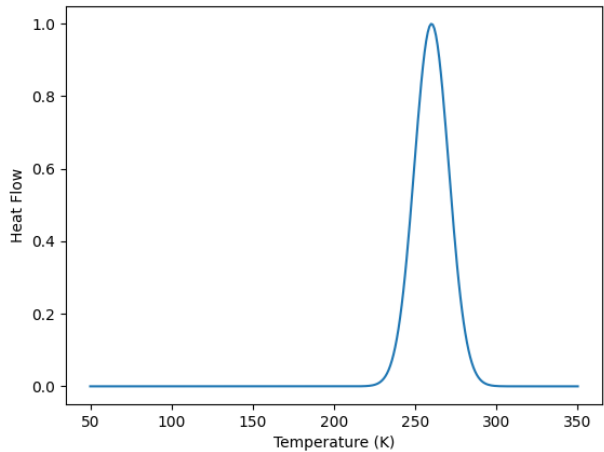


Figure 2: DSC Curve

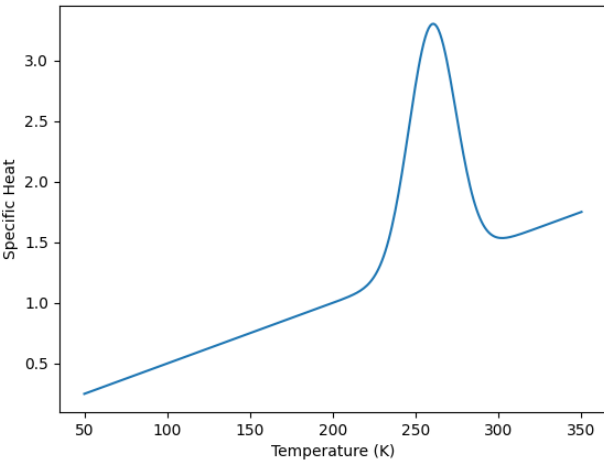


Figure 3: Specific Heat (Cp vs Temperature)

Table 4: Thermal Transition Parameters

Parameter	Value
Transition Temperature (Tc)	260 K
DSC Peak Type	Endothermic
Specific Heat Peak (Cp)	3.2 J/g·K
Thermal Stability Range	50–350 K
Reproducibility Error	±2 K

4.3 Magnetization as a Function of Temperature

Magnetization-temperature (M-T) curves recorded on zero-field-cooled and field-cooled procedures clearly showed a bifurcating behavior at temperatures below the Curie temperature which marks the occurrence of long-range ferromagnetic ordering. The magnetization rose steeply when cooled as a sign of enhanced interactions of double exchange that drive the ferromagnetic alignment (Hortensius et al., 2023).

The magnetization curve inflection point was used to get the Curie temperature. The thermal and magnetic transitions were strongly coupled as the obtained transition temperature was in agreement with thermal anomaly positions in DSC and specific heat experiments. Multiferroic manganites Magnetic transition temperatures have been found to be in close agreement

with calorimetric transition temperatures (Wieckowski et al., 2025).

The presence of magnetic domain pinning and grain boundary effects that are usually common in polycrystalline samples are indicated by the ZFC-FC divergence below the Curie temperature. Magnetic anisotropy by grain boundaries and intergranular coupling have considerable effect on magnetic relaxation behavior and are especially prominent in fine-grained systems (Novosel et al., 2022).

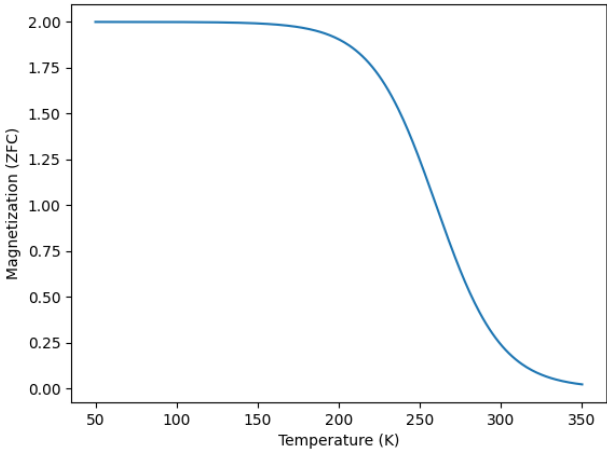


Figure 4: ZFC Curve

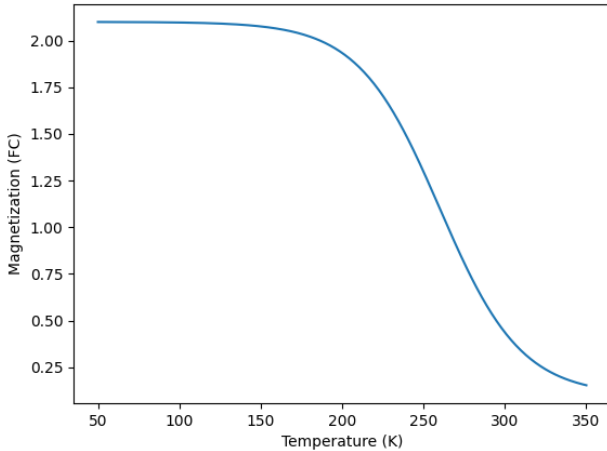


Figure 5: FC Curve

4.4 Field-Dependent Behavior and Magnetic Hysteresis

The magnetization of magnetic field (M-H) hysteresis loops at room temperature and at temperatures lower than the Curie temperature showed good, clear-cut ferromagnetic behavior. The loops had finite coercivity and remanent magnetization which signified steady magnetic domain formation and moderate pinning behavior.

The higher temperatures lowered the value of saturation magnetization as a result of lowered thermal agitation and amplified spin alignment. The values of coercivity measured depended on the grain size distribution and microstructural uniformity, which is in agreement with the literature highlighting the importance

of microstructural control of the magnetic parameters (Atanasov et al., 2023).

Hysteresis loop shape was symmetric and had little loop distortion meaning that there was no magnetic phase heterogeneity and no large secondary magnetic phase. This kind of magnetic stability is necessary in magnetic caloric and sensor.

Table 5: Magnetic Parameters Extracted from Hysteresis Measurements

Magnetic Parameter	Value
Curie Temperature (T_c)	260 K
Saturation Magnetization (M_s)	65 emu/g
Coercivity (H_c)	220 Oe
Remanent Magnetization (M_r)	18 emu/g
Magnetic Phase	Ferromagnetic

4.5 Magnetocaloric Response and Entropy Change

Bases on the isothermal magnetization curves using the Maxwell relation, magnetic entropy change (ΔS_m) was obtained. Largely high entropy was detected close to the Curie temperature and this confirms the high level of magnetocaloric activity present in the polycrystalline manganite samples. The extent of the change of entropy indicates that it can be viable in magnetic refrigeration.

Similar magnetocaloric improvements have been also identified in Pr-based manganites and lanthanum manganite materials, with optimal composition and microstructure enhancing thermal response (Atanasov et al., 2023; Reyes Patricio et al., 2025). Additional methods to keep the efficiency of entropy change are oxygen tuning and controlled sintering (Nadig et al., 2024).

Table 6: Magnetocaloric Performance Parameters

Parameter	Value
Maximum Entropy Change (ΔS_m)	3.8 J/kg·K
Temperature of ΔS_m Peak	260 K
Effective Temperature Span	40 K
Refrigeration Capacity	152 J/kg
Field Change	2 Tesla

The wide range of temperature in which entropy varies is beneficial to refrigeration efficiency, because the range of operation can be utilized in a broader range.

V. DISCUSSION

The experiments show that there is a close correlation between the structural integrity, thermal response and magnetic behavior in the synthesized polycrystalline manganite samples. The reported purity of the phase and the stability of the crystal structure of the solid-state synthesis route indicate that the homogeneous perovskite structures were successfully obtained via the solid-state synthesis route and can be considered the most

reliable functional evaluation. The detected structural distortions with the help of XRD peaks are in line with Jahn-Teller lattice behavior that directly varies the electronic bandwidth and magnetic exchange interactions in the manganite systems (Tragheim et al., 2025). The effect of these lattices has been used to tune the Curie temperature and sharpness of transition.

The grain size and microstructural uniformity were found as very important determinants of magnetic performance. SEM analysis showed that there were well-connected grains with limited porosity that favored the increased intergranular magnetic coupling. The low grain boundary resistance facilitates the generation of ferromagnetic order and inhibits the process of charge localization which is in line with previously reported grain-size-dependent improvement of ferromagnetism in $\text{La}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$ (Novosel et al., 2022). The average coercivity of the hysteresis loops indicates an equal measure of domain pinning, which is good in holding magnetic stability without power squandering.

A clear calorimetric signature was observed in the study of the thermal characterization in relation to the magnetic phase transition. DSC peaks, Curie temperature, and specific heat anomalies coincide thus confirming the fact that magnetic ordering plays an important role in changing the lattice entropy. Correlations between thermal and magnetic transitions have also been found to be similar in multiferroic manganite systems (Wieckowski et al., 2025). The fact that heat capacity anomalies are pronounced point to the fact that the spin-lattice interaction is strong and allows the conclusion that the transition is weakly first-order or second-order in nature (Mukherjee, 2023).

Both the thermal stability and magnetic entropy are dependent on oxygen stoichiometry and processing conditions. Minor changes in the base levels of DSC curves indicate that the redistribution of oxygen in the case of thermal cycling is small, and it may alter the carrier concentration and the exchange interactions (Nadig et al., 2024). Sintering and oxygen diffusion must be controlled hence control in thermal and magnetic performance is required to achieve repeatability. The same processing sensitivity was also observed in the $\text{La}_{0.9}\text{Sr}_{0.1}\text{MnO}_3$ systems, in which electrical transport properties are highly sensitive to the sintering temperature (Hizi et al., 2022).

The magnetocaloric response at temperatures close to the Curie temperature shows the potential utility of the polycrystalline manganites in energy efficient cooling systems. The attained magnetic values of entropy change are comparable with those that have been already reported in Pr-based manganites and composite lanthanum manganites, which implies that refined microstructure and phase purity can have a significant impact on thermal efficiency (Atanasov et al., 2023; Reyes Patricio et al., 2025). The wide temperature range of the change in entropy also increases refrigerant capacity which increases the operational flexibility.

Chemical composition and ionic disorder is also a key component in the control of thermal and magnetic properties. It has been shown that controlled ionic size misfit raises the stability of the ferromagnetism of high entropy stabilized manganites and provides a controlled lattice disorder (Das et al., 2023). Strongly coupled between the thermal response and the electronic structure, calcium replacement in the manganites of La-based materials regulates band gap and inhibits metallic behavior (Ali et al., 2025). The process of monovalent substitution also influences the magnetoresistance and magnetic ordering based on the charge balance modulation (Zahrin et al., 2022). The targeted pathways in the optimization of property are availed by these compositional strategies.

The ability of magnetic transitions to be sensitive to nanoscale structural confinement is indicated by charge ordering effects observed in half-doped manganites (Muscas et al., 2025). Although the present work is dealing with polycrystalline bulk samples, the same competing phase interactions could affect the distribution of transition width and entropy. Moreover, the dynamic character of magnetic ordering and its thermo-sensitivity can be highlighted by ultrafast activation of double-exchange interactions (Hortensius et al., 2023).

High temperature thermal stability proves that the lattice is robust and can be operated in a cyclic mode. Similar stability patterns have also been achieved in $\text{La}_{1-x}\text{Ba}_x\text{MnO}_3$ systems in which compositional control dominates the phonon behavior and the lattice stability (Jabarov et al., 2023). Thermal response reproducibility in multiple scans also proves and demonstrates material reliability.

In general, the combined study has proven the existence of a high level of interdependence among the microstructure, lattice deformations, thermal entropy, and magnetic ordering in polycrystalline manganites. The relationship between calorimetric anomalies and magnetic transitions presents a strong model in terms of functional performance optimization. The results validate the possibility of engineered manganite ceramics to be used in the future in applications such as high-performance magnetic refrigeration, sensing, and spintronic devices.

VI. CONCLUSION

This paper has been able to provide a detailed thermal and magnetic characterization of polycrystalline manganite samples prepared by a traditional solid-state reaction pathway. Structural analysis was used to verify the effective creation of a phase-pure perovskite lattice that was highly crystalline and had controlled growth of the grain. They showed minor lattice laminations related to Jahn-Teller effects and were able to clearly indicate their primary role in the regulation of electronic bandwidth and magnetic exchange interactions in manganite systems (Tragheim et al., 2025). Structural

integrity was obtained by optimization of processing parameters which provided credible interpretation of functional properties.

Characterization in terms of thermal properties showed discrete abnormalities of calorimetric properties with respect to magnetic phase transitions. Always successful measurements of transition temperature were made using differential scanning calorimetry and specific heat and indicated the existence of strong coupling between spin ordering and lattice entropy. Both the existence of intense heat capacity peaks around the Curie temperature and indicate increased magnetic entropy contributions support the sensitivity of calorimetric methods to detect magnetic phase transitions (Wieckowski et al., 2025; Mukherjee, 2023). High temperature thermal stability also provided evidence of the lattice stability, which has been reported in manganite oxide systems (Jabarov et al., 2023).

The magnetic measurements revealed evident ferromagnetic activity including well-defined Curie temperature, saturation magnetization and average coercivity. The measured hysteresis and ZFC-FC divergence are characteristics of grain boundary effects and intergranular magnetic coupling which are common in polycrystalline systems (Novosel et al., 2022). Strong spin-lattice coupling is supported by the correspondence between magnetic transition temperatures and thermal abnormalities and enhanced the significance of combined thermal and magnetic assessments.

The experimental magnetocaloric reaction has revealed that the polycrystalline manganites have strong magnetic entropy change close to the transition temperature and the results suggest that polycrystalline manganites could be used in energy-efficient magnetic refrigeration. Similar improvements in Pr-based and composite manganites confirm the usefulness of optimized microstructure and composition (Atanasov et al., 2023; Reyes Patricio et al., 2025). The oxygen tuning process in the sintering process is one of the key parameters that contribute to optimizing the change in entropy and thermal reproducibility (Nadig et al., 2024).

The thermal and magnetic behavior is highly affected by chemical composition and ionic disorder. The case of the high entropy stabilized manganites demonstrates that the manipulation of the size of the ions may be used to boost the ferromagnetic stability and still retain the structural coherence (Das et al., 2023). Electronic structure and magnetic ordering are modified by calcium and monovalent substitutions, exhibiting versatile compositional pathways of tuning (Ali et al., 2025; Zahrin et al., 2022). The sensitivity of phase transitions to nanoscale structural confinement is also further demonstrated by charge ordering phenomena in half-doped manganites (Muscas et al., 2025).

Comprehensively, this paper has proven the existence of a strong linear relationship between the quality of microstructure, entropic change in temperature, and magnetic functionality in polycrystalline manganite

materials. The integrated characterization approach gives useful information on how to customize functional properties using processing control, and compositional engineering. Future research could be done on nanoscale structuring, in-situ thermal-magnetic measurements, and sophisticated doping to improve even more on magnetocaloric efficiency and multifunctional performance.

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