Direct Imaging of Dynamic Glassy Behavior in a Strained Manganite Film

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Glassy states are of crucial importance for both fundamental science and applications[1, 2]. Despite being one of the oldest problems, the intriguing dynamics close to the vitrification point are still poorly understood. Part of the difficulty arises from the traditional bulk measurements being hard to interpret. Here, we performed a microscopic study of the strain-induced[3, 4] glassy behavior in epitaxial Pr0.55(Sr0.75Sr0.25)0.45MnO3 (PCSMO) thin films[5–8] using a novel microwave impedance microscopy (MIM)[9, 10]. The strong MIM contrast between spatially coexisting ferromagnetic metallic (FM-M) and charge-ordered insulating (CO-I) phases[11] reveals the microstructure of the slow relaxation behavior. Randomness of the phase separation from different cooling processes and cross-correlation of time-dependent images provide direct evidence of the non-ergodic and freezing behaviors. Our data also reveal a connection between underlying lattice strains and the glassy kinetics at mesoscopic length and laboratory time scale, demonstrating the strikingly extended hierarchy of scales in glassy physics.

A conventional glass or structural glass is formed by rapid cooling of a viscous liquid, resulting in a disordered solid with no crystallinity. Similarly, the lack of symmetry can be found in a disordered magnet or the so-called spin glass, in which spins are frozen in random directions. While analogy can be drawn between these two glassy systems, the lack of a true ground state in spin glasses distinguishes them from the conventional counterpart. Driven by the desire to understand colossal magnetoresistance, the glassy behavior of the perovskite manganites has attracted great recent attention[7, 12–24]. The manganite glass is unique because atomic scale interactions lead to the spatial separation of two phases with distinct electrical and magnetic properties at much larger length scales. Despite substantial progress, the nature of this phase separated (PS) glassy state remains poorly understood, with opinions varying from conventional glass[23], spin glass[22], and a new glass family[13]. Uncertainty in previous work largely stems from the fact that macroscopic measurements, which average over the entire sample, are hard to interpret. To make progress, nanoscale imaging[11, 23–25] of the glassy phase in manganites is desired to yield deeper insights at length and time scales not addressed before.

In addition to the intertwined charge, spin, and orbital degrees of freedom, complexity in manganites also arises from the cooperative lattice strains between the pseudo-cubic FM-M and distorted CO-I phases[3, 4]. Recent advances in thin film epitaxy of complex oxides provide a viable method to control such strain effects. Here, we took thin film Pr0.55(Ca0.75Sr0.25)0.25MnO3 (PCSMO) with y = 0.25 as a model manganite system. In the bulk compound, PCSMO at this composition exhibits the bi-critical behavior[8] in that at the same phase transition temperature, whether the high temperature CO-I to PM-I (rather than FM-M) phase is barely discernible at T ≈ 220 K. The FM-M state, on the other hand, can be easily recovered by the application of a small field warming (FW) after zero-field cooling (ZFC) is reached (Fig. 1a, dashed line). Interestingly, the T-dependent magnetization (Fig. 1b) taken during various field warming (FW) after zero-field cooling (ZFC) is small at low temperatures, increases rapidly at T ≈ 20 K, and decreases again at T ≈ 120 K, indicating that the FM-M state is favorable within this temperature range. These observations suggest the energy proximity between CO-I and FM-M phases, a prerequisite for PS glass. Microscopic PS of manganites can be readily observed by MIM[11], which functions by sending a 1 GHz microwave signal into a coaxial cantilever probe[26] and measuring the nanoscale sample impedance. The real
FIG. 1: Transport measurements and MIM measurement. a, Resistivity of a fully strained PCSMO film on LSAT substrate (red) and a relaxed film on CTO/STO substrate (black). The blue dash line is taken during FW after ZFC with $B = 1$ T. b, ZFC-FW magnetization of the LSAT film under different B fields. c, Schematic of the MIM measurement on the PCSMO film and the equivalent circuit of the tip-sample interaction. When the tip sits on the MnO$_2$ particles, the MIM signal is the lowest due to an additional $C_{MnO_2}$ in series with the sample impedance. In the FM-M regions, the low $R_{PCSMO}$ of the metallic domain shorts the CPCSMO, which results in a higher MIM signal than that in the CO-I region. A larger FM-M domain results in larger coupling to the substrate, thus appears brighter (higher MIM signals) than a smaller domain.

and imaginary parts of the MIM outputs contain local conductivity information of the sample down to 50nm. Fig. 1c shows a schematic of the MIM probe on top of the 30nm PCSMO film and the equivalent lumped-element circuit. Many MnO$_2$ precipitates[27], which are always insulating within our temperature and field ranges, are present on the sample surface and conveniently serve as landmarks. Other than these defects, the surface is nearly atomically flat. For this study, the MIM response in the imaginary channel, which is proportional to the tip-sample capacitance[9, 10], is sufficient to identify the MnO$_2$, CO-I and FM-M regions; and they appear in the false color maps as black, red, and yellow, respectively.

We first studied the microscopic arrangement of the PS glass as a function of the cooling rate at $B = 0$. Before each data point, the sample was warmed up to 250K, above the charge-order temperature $T_{CO}$, to erase any prior history. Two processes, a fast cool down (FCD) at 8 K/min and a slow cool down (SCD) at 0.3 K/min, were employed to reach specific temperatures under which the images were taken. Fig. 2a and 2b (2c and 2f) show representative FCD and SCD images at 12K (50K), respectively. Without any data analysis, it is already striking to see the substantial FM-M fractions in the nominally insulating film. Indeed, careful image analysis (Fig. 2i) shows that the FM-M states can occupy up to 40% of the sample when cooling at zero field. Considering the minute magnetization obtained in the macroscopic measurement (Fig. 1b), the only plausible explanation is that these FM-M clusters are macro-spins that freeze at random orientations[21].

One central assumption in glass physics is non-ergodicity[2], i.e., the inability of the system to traverse the available phase space. The summary plot in Fig. 2i shows systematically lower FM-M fractions after SCD than FCD. Both curves develop a maximum at $T \approx 50$ K, with the one in SCD being less prominent. Similar behavior were observed in the magnetization data under 1.2T field[19] (Supplementary Information S2) but less distinction between the FCD and SCD, possibly caused by the incoherence of the spins. Interestingly, while the FM-M domains occupy about the same area after two identical SCDs, the MIM images are microscopically different. In Fig. 2c and 2g, we extract the FM-M domains and overlay them for comparison. At 12K, about 50% of the FM-M domains emerge at different locations and the number is over 60% at 50K. The overlapped domains are likely pinned by intrinsic defects[11], but the randomness is a clear evidence of multiple free-energy configurations.

As mentioned before, the substrate-induced elastic strain plays an important role in the glassy behavior. The autocorrelation analysis of the SCD images shows the tendency of FM-M domains to elongate along the [110] axis of the substrate. Such a lattice effect may also explain the cooling rate difference in Fig. 2i. Fig. 2j illustrates the crystal structure of the PCSMO film in the (001) plane. Both PM-I and FM-M phases have similar cubic structures, while the CO-I phase undergoes orthorhombic distortion and can be stabilized by strain-induced structural transition[? ]. As temperature decreases to around 50 K, FM-M becomes energetically comparable to CO-I. During the FCD, the system does not have enough time for structural changes; thus many regions with undistorted FM-M domains are found. For the SCD, more areas switch to CO-I, making it harder to transition to FM-M at lower temperatures.
FIG. 2: Cooling Speed Dependence. a–c (e–g) From left to right, MIM images at 12 K (50 K) after FCD, after two SCDs, and overlays of the domains from the two SCDs, respectively. d and h, Autocorrelation analysis of the SCD I images, which illustrates the preferred alignment of FM-M domains along the [1-10] axis of the substrate. i, FM-M fractions of the two processes as a function of temperature. j, Structural changes during the cooling process. Both PM-I and FM-M phases both have an undistorted pseudo-cubic structure, while the structure of CO-I is distorted orthorhombic. All scale bars are 2 μm.

FIG. 3: Relaxation at different temperatures. a, MIM images after arriving at 12, 25, 50 and 70 K, respectively after FCD, and b, images after waiting for several hours. At 12 K, no change was observed (frozen). At T > 20 K, the domain are dynamic with increasing FM-M domain fraction at 25 and 50 K and decreasing FM-M domain fraction at 70 K. c, Cross-correlation coefficient $r_{xy}$ as a function of temperature, showing a sharp jump of $r_{xy}$ at $T_g \approx 20$ K. The inset summarizes the FM-M fraction before (solid) and after (empty circle) waiting for 4 hours. All scale bars are 2 μm.

Another characteristic of glass is the divergence of relaxation time approaching $T_g$, the glass transition temperature. To investigate this property microscopically, we first prepared the sample by zero-field FCD to a certain temperature, and then took repeated scans on the same area at this T. Comparisons between MIM images immediately after FCD and several hours later, a waiting time much longer than the cooling, are shown in Fig. 3a and 3b. At the first glance, the results for T > 20K are perplexing. After 4 hours, the FM-M areas increased for T = 25K and 50K and decreased for 70K. And the long waiting time after FCD did not bring the system towards the SCD state (Fig. 3f). More importantly, many FM-M domains appeared at different locations even when the change in FM-M percentage was small. In other words, a simple measure of the areal frac-
tion (inset of Fig. 3c) cannot capture the dynamics here. The microscopic imaging, on the other hand, allows us to calculate the cross-correlation coefficient \( r_{xy} \) before and after the waiting time. As shown in Fig. 3c, \( r_{xy} \) is small (≈ 0.5) at high temperatures but rapidly rises towards 1 below 20K, a vivid demonstration of the freezing of the PS glass at this temperature. Indeed, the transition from a dynamic state to a frozen state at \( T_g \approx 20 \text{ K} \) is also exemplified by the 12K image showing no change after 18 hours (Supplementary Information S7). We further conjecture that the kinetic barrier formed during the glass transition is responsible for the reentrant behavior in Fig. 1b, with the sudden drop of magnetization around \( T_g \) as due to glass freezing effects. This effect is earlier reported in another manganite compound, and is attributed to a strain glass transition[16–20].

In Fig. 4a (Supplementary Information S8), we further investigate the dynamics of the PS glass by applying a B-field and studying the relaxation after removing this external stimulus[15]. The sample was again prepared by FCD to the desired \( T \) before turning on the B-field. A field of 2.4T induced a significant portion of FM-M phases at all temperatures. Immediately after its removal, however, very different behaviors occurred at various temperatures. For \( T < T_g \), the PS configuration was frozen after switching off the field and remained so after 18 hours. Since the magnetization dropped almost by half (Fig. 4b) upon field removal, the interpretation is again the randomization of spin orientations. At 50 K, no obvious change was observed right after turning off \( B \), while the insulating regions increased after about a day. Again, the CO-I or FM-M phases in Fig. 2 and 3 preferentially aligned along the [110] direction. The relaxation was much faster at 70K, where large changes happened right after the field removal and continued in several hours. For high enough \( T = 120\text{K} \), the relaxation back to the zero-field state was faster than our imaging time so little variation could be seen for another hour of waiting. Fig. 4c summarizes the measured FM-M fraction throughout this process. Assuming logarithmic time dependence, we can extract the relaxation time as a function of temperature (Supplementary Information S6). Fitting such plot (Fig. 4d) with the Arrhenius equation, we obtain a rough estimate of the activation energy barrier \( \Delta E = (1.3 \pm 0.9) \times 10^2 \text{ K} \) in the dynamic regime.

As a final remark, the PS glass around 50K appears to be an intrinsic mix, with neither FM-M nor CO-I being the obvious lower energy component to which the sample will gravitate. After hours of waiting, the system may develop more FM-M after FCD (Fig. 3) or more CO-I after the field removal (Fig. 4). This dual behavior suggests that the two competing phases have very similar energies, with relative fraction determined by higher order effects such as interactions among the macro-spin clusters. While the underlying charge, spin, and lattice physics is of atomic scale, the interactions among them lead to clusters and correlation lengths as long as micrometers. And the anisotropic strain clearly plays a key role in this mesoscopic glass. It is likely that this large span of length scales is coupled with the huge time span ranging from sub-picosecond electronic interaction to the laboratory scale of hours or days.

In summary, microscopic imaging of PCSMO directly visualizes the non-ergodicity and freezing behavior, key hallmarks of a glass state. It also suggests that this system contains both conventional glass and spin glass characteristics, with the former connected with Ising-like mixture of FM-M and CO-I phases and the later connected with random spin orientation among the FM-M domains. This unifies the conflicting reports on the nature of manganite glass. The rich glass physics is also shown to be tied to the lattice strain. Such knowledge would not be possible without the ability to image the system with high spatial resolution, strong contrast, good stability, and accurate registry for time-dependent studies.
FIG. 4: Relaxation after removal of B-field. a, MIM images taken at $B = 0$ T (before), $B = 2.4$ T, $B = 0$ T (after) and after several hours at the same condition (After + Time) at $T = 12, 50, 70, \text{and } 120$ K, respectively. For $T < T_g$, no change is observed after $B$ is switched off. b, Magnetization as a function of time. The B field is applied at time zero and removed after 4 hours. The arrows indicate where the MIM images were taken. c, FM fraction before (red) at $B = 2.4$ T (blue), after (green) and 3 hours after (yellow). d, Relaxation time as a function of temperature. The characteristic energy $\Delta E = (1.3 \pm 0.9) \times 10^2$ K is estimated. All scale bars are 2 µm.

Materials and Methods

Pr$_{0.55}$(Ca$_{0.75}$Sr$_{0.25}$)$_{0.45}$MnO$_3$ (PCSMO) films with $y = 0.25$ were grown on (110) (LaAlO$_3$)$_0.3$(SrAl$_{0.5}$Ta$_{0.5}$O$_3$)$_{0.7}$ (LSAT) and CaTiO$_3$/SrTiO$_3$ (CTO/STO) substrates by pulsed laser deposition. The thicknesses of the films are about 40 nm. Resistivity, magnetization measurements, and reciprocal space maps for both films are provided in Supplementary Information S1. The magnetization of the sample was measured with a SQUID magnetometer (Quantum Design MPMS). The relaxed film on CTO/STO substrate behaves like bulk PCSMO, indicating high quality film growth.

MIM images were taken in a cryogenic MIM setup described in reference [9]. MIM functions by sending a microwave signal at 1 GHz into a micro-fabricated coaxial probe. Low input power $\approx 1\mu$W is used to avoid disturbing the sample. The reflected signal contains local electrical information of the sample and is demodulated at the same frequency, giving MIM outputs. The spatial resolution is about 50 nm. One MIM image takes about 30 minutes to an hour. Methods for overlaying images and calculating cross correlation coefficient are described in Supplementary Information S3. Procedures for obtaining the areal fraction of the FM-M domains and autocorrelation analysis are described in the Supplementary Information S4 and S5.

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Author contributions

Z. Sheng and M. Nakamura grew the PCSMO sample. W. Kundhikanjana performed the MIM measurements and the data analysis. Y. Yang, Q. Tang, K. Zhang, and X. Li fabricated the MIM probes. W. Kundhikanjana, K. Lai, and Z.-X. Shen wrote the manuscript. All authors participated in the scientific discussion during the manuscript preparation.
**Supplementary Information**

**PCSMO films on LSAT (110) and CTO/STO (110) substrates**

Two high qualities PCSMO films growth on LSAT and CTO/STO substrates are characterized by X-ray reciprocal mapping, resistivity and magnetization measurements. S1(a), Reciprocal space map of the PCSMO film on LSAT. The (222) and (310) peaks of the film align with the LSAT substrate. The widths of diffraction peaks of film along lateral direction, corresponding to the omega scan of crystal, are comparable to those of substrates suggesting a coherent growth and high quality film. S1(b), ZFC-FW resistivity curves in different magnetic fields also show the reentrance of the insulating state. S1(c), Magnetization as a function of temperature for ZFC-FW (black), FC (green) and, FC-FW (red). S1(c) (inset), Resistance as a function of magnetic field after ZFC to 12 K (blue) and 120 K (magenta). PCSMO film on LSAT substrate is quite different from a bulk sample.

A PCSMO film on CTO/STO substrate behaves rather similar to the bulk sample, which is different from the film on LSAT. S1(d) shows reciprocal space map of the PCSMO film on CTO/STO shows the large deviation from the LSAT substrate. S1(e), Resistivity measurements show the film on the CTO/STO substrate is metallic at low temperature. S1(f), Magnetization curve taken during ZFC-FW, FC, FC-FW are the same. No reentrance behavior is seen. The lattice constant of CTO in a cubic convention is 3.826 Å, closer to that of the PCSMO lattice[8], while the lattice constant of LSAT is larger, 3.869 Å. We called the film on CTO/STO a relaxed film and on LSAT a strained film. From the basic characterizations, we can conclude that the tensile strain from the LSAT substrate favors the CO-I phase, induces strong competition between the CO-I and FM-M phases, and is responsible for the glassy behavior. The bulk-like behavior in a CTO/STO film also confirms the high-quality growth.

The PCSMO film in this study contains MnOx precipitates on the sample. We emphasize that the presence of MnOx particles does not imply a low quality film since the characterizations mentioned above indicate a high quality film growth. In addition, attempting to remove these particles by tuning the growth condition such as such as oxygen pressure and laser spot size on the target resulting in a film with different stoichiometry. This problem is common in film growth of strongly-correlated materials, such as in cuprate films [28, 29] and manganites[27, 30]. The stoichiometry of PCSMO film with doping level \( y = 0.25 \) is very important for the critical feature, and thus the glassy behavior. Therefore, we adopted the correct stoichiometric sample with MnOx precipitates for this study. We emphasize that the MnOx particles give distinct MIM response and does not hinder the study of the phase-separated region as mentioned in the caption of Fig. 1(c). They are also excluded from the statistic, when calculating the FM-M fraction (see S4 for details).

**Observation of reentrance of CO-I state in FM-M fraction and virgin magnetization**

S2(a), FM-M fraction extracted from the MIM images as shown in Fig. 2a in the main text. S2(b), Temperature dependence of magnetization taken by a virgin method. The virgin magnetization was measured by cooling down to the desire temperature, turning on a \( B = 1.2 \) T field to measure the magnetization, then thermal cycling before the next data point, similar to the way the MIM images were obtained. Peaks at \( T = 50 \) K were observed for both fast (red) and slow (blue) cool downs with a smaller peak

![FIG. S1: PCSMO films on LSAT (110) (left) and CTO/STO (110) (right) substrates.](image)

![FIG. S2: a, Reentrance of CO-I state in FM-M fraction. b, Virgin magnetization.](image)
for the slow cool down curve. Due to application of the B field, the magnetization curves are broader than the FM-M fraction curves extracted from MIM images. Both the MIM and the virgin magnetization measurements reflect the reentrant behavior of the CO-I phase. The cooling speeds for fast and slow cool down in the magnetization measurement were 10 K/min and 2 K/min respectively.

**Overlap of the FM domains, and cross correlation coefficient**

**S3(a),** MIM images at 12 K after the two slow cool downs. The images were first aligned by optimizing the standard cross-correlation coefficient of the raw images. We extracted the black-and-white images showing only the FM-M domains before overlaying them on top of each other. **S3(b),** Image at 70 K from Fig. 4 of the main text. Overlaying the images of the FM-M domains in the absence of B and after B is turned on clearly shows both the disappearance of existing domains and the formation of new domains. The cross correlation coefficient \( r_{xy} \) shown in the main text is calculated from the black-and-white images. The white region is given the value 1 and the black region the value -1. The \( x_{eff} \) is equal to the sum of the dot product of this two images, divided by the size of the image.

![Overlap of FM domains](image)

**Method for extracting the FM-M fraction**

The areal fraction of the FM-M domains was obtained by the following procedure.

1. Divide the MIM image into 10 × 10 pixels images.
2. Find a local maximum corresponding to the domains of each images.
3. Put the images back together to obtain the image in **S4(b).**
4. Domains smaller than 20 pixels in size were considered noise and were removed resulting in the image in **S4(c).**
5. Using the same procedure, we extract the area covered by MnO₂ particles **S4(d),** where states underneath are unknown.
6. The FM-M fraction was obtained by counting the number of the white pixels in c, and divided by number of the black pixels in **S4(d).**

![Method for extracting FM-M fraction](image)
Autocorrelation analysis

The autocorrelation analysis presented in the main text was obtained with the following method. The metallic domains were extracted using a crude threshold value, resulting in the black-white image in S5(b). Autocorrelation image is shown in S5(c). S5(d), Plot of the linecuts along the vertical (blue) and horizontal (red) lines of the autocorrelation. The characteristic length along the [1¯10] and [001] axes were obtained by fitting exponential functions to the vertical and horizontal linecuts.

Time dependence FM-M fraction and relaxation time.

Although most transport studies (for example Ref.[14]) prefer fitting magnetization data with a logarithmic function, we choose a exponential function in our fit. This is because the data points from our scans are taken an hour apart, which is a coarse time scale compared to the transport data. The scanning data captures a slow relaxation process and is suitable for an exponential fit.

S6(a) plots the FM-M fraction as a function of time. The solid lines are exponential fits using

\[ M(t) = M_0 + Ae^{-(t-t_0)/\tau} \]  

The relaxation time \( \tau \) is extracted and is plotted as a function of temperature in S6(b). We fit the temperature dependence of the relaxation time using an Arrhenius activation barrier equation.

\[ \frac{1}{\tau} = Be^{-E/kT} \]

The average activation barrier obtained from the fit \( \Delta E = (1.3 \pm 0.9)^2 \text{ K} \).

Additional Images

FIG. S6: a, FM-M fraction as a function of time. b, Relaxation time as a function of temperature.

FIG. S7: Frozen and unfrozen glass. The top and the middle rows are black-and-white image showing the FM-M domains after fast cool down and after waiting for 4 hours (data in Fig. 3). The bottom row are images showing the overlap of FM-M domains. The white, red and blue indicate the domains, which are unchanged, newly created, or have disappeared after waiting. All scale bars are 2 \( \mu \text{m} \).
FIG. S8: Due to limited space only half of the MIM images were shown and image at 100 K was omitted in Fig. 4. The full images are shown here.

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