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ABSTRACT

Four decades since the concept of polyamorphism was introduced by [L. S. Palatnik (1909–1994), Fiz. Nizk. Temp. **25**, 400 (1909)], numerous investigations proved its presence in a broad variety of nonmagnetic short-range ordered materials, like structural, metallic, a-metallic, inorganic molecule, orientational, electron glasses, water, ice, carbons, and others. It was manifested by phase transitions between amorphous states as a function of the quench condition and under compression, mediated by long-wave fluctuations of an order parameter. There has been much recent discussion given to the phenomenon of polyamorphism where distinct, different states of amorphous liquids and solids are observed as a function of density. The outstanding contribution of the recently late [A. Sella, *et al.* (1956–2022), Nat. Mater. **21**, 490 (2022)],[2] in the field should be recognized here. Underlying this phenomenon is the possibility of a first-order liquid-liquid phase transition driven by the density and entropy differences between the two amorphous phases. Magnetic boost of multilayer graphene under pressure was also recently discovered. Their famous spin counterparts, such as spin liquid, spin ice, and spin glass have been less studied at this end despite numerous similarities, registered so far. Taking that in mind, for further polyamorphism platform development, we demonstrate the signatures of phase transitions in spin glass, driven by a magnetic field, and eventually, a novel type of polyamorphism, the spin-glass one.

In Memorium: Lev Samoilovich Palatnik (26 April 1909–04 June 1994)

Paul Francis McMillan (3 June 1956–2 February 2022)

1. INTRODUCTION

The concept of polyamorphism in a state of the matter without long-range order, as a counterpart to polymorphism of ordered phases, was first introduced in 1981 in a seminal work[3,4] and references therein) of outstanding scholar and scientist, the founder of thin-film science and technology in our country, professor at the Kharkiv Polytechnic Institute, Lev S. Palatnik, entitled "*Polyamorphism and substructure of short-range order in amorphous boron films*". Here the structure and substructure of boron amorphous films have been studied in detail. Two amorphous phases of short-range ordered icosahedrons were revealed as a result, and attributed to the boron vacancies formation. This work is not always cited in a growing number of research and review articles in the field, as it is hard to access, perhaps. Here, we attempt to fill this gap. The concept of polyamorphism sets a framework for generalized consideration of phase transitions in amorphous media brilliantly developed by recently deceased Paul McMillan (Refs. 2 and 4 and references therein). Four decades later, the superconducting quantum phase transition (QPT)[5] has been found in amorphous WSi thin film[6] and electronic phase evolution under pressure in van der Waals compound $FePS_3$ (magnetic graphene),[7,8] molecular framework $Ni(NCS)_2$,[9] and displacive ferroelectric.[10] Both transitions are unexpected, where they are, but realized in the presence of randomness and fluctuations under external action.[11] The slow relaxation to equilibrium states, typical of such exotic phase transitions, is intuitively in relevance with the hidden order and fluctuations. They were observed for both, quantum and polyamorphic phase transitions.[12–19] Meantime, the convincing experimental evidence for polyamorphism has been reported for many *liquids*,[20,21] including water,[22–127] amolecular

liquid,[28] sulphur melt,[29] $A_{l2}O_3$–Y_2O_3,[30] phase-change alloys,[31–33] germanium,[34,35] GeTe and GeSe,[36] silicon,[37–40] phosphorus,[41] amorphous ice.[42–44] The concept of polyamorphism facilitated the unifying description of a fluctuation mediated[45–49] and references therein) annealing in glass, first observed well before.[50] Since then, a diverse variety of *structural glasses*, glass-forming liquids,[51–57] and amorphous films[58,59] have manifested signatures of polyamorphism, including yttria–alumina liquids,[60] silica,[61–63] SiO_2,[64] chalcogenide,[65] inorganic molecular,[66] borosilicate,[67] AuCuSi alloy,[68] ametallic glass.[69] Above mentioned research has demonstrated, similar to Refs. 1, directional and open (such as tetrahedral) coordination environments as a precursor of polyamorph transition. Its manifestation in dense *metallic glasses*[70] was, therefore, a surprise, explained, in particular within a polycluster approach.[71,72] A lot of metallic glasses[73–77] and references therein) revealed polyamorphism under pressure. Among them, there are Ce-based[76,77] ($Ce_{75}Al_{25}$,[78] As_2Te_3,[79] Lantanide-based,[80,81] Yb-based,[82] main-group metallic glass,[83] high entropy,[84] ultrastable[85] metallic glasses. It is reported an in situ X-ray diffraction observation of a pressure-induced transition between two distinct amorphous polymorphs in a $Ce_{55}Al_{45}$ metallic glass. The large density difference observed between the two polyamorphs is attributed to their different electronic and atomic structures, in particular the bond shortening revealed by ab initio simulation of the effects of *f*-electron delocalization.

As an obvious example, the pressure-induced polyamorphic transformation from the low-density to the high-density state was found in lanthanide-based metallic glasses (Fig. 1). These polyamorphic transformations demonstrate the electronic structure inheritance of lanthanide atoms in metallic glasses. So far, polyamorphic phase transitions in lanthanide-containing metallic glasses have been observed only in lanthanide-solvent metallic glasses. A question arises whether polyamorphism is also possible in lanthanide-solute metallic glasses.[81] In collaborative team from Harbin Institute of Technology, HPSTAR, and Argonne National Laboratory investigated this puzzle using an advanced synchrotron X-ray technique. A pressure-induced transition between two distinct amorphous states was observed in $La_{43.4}Pr_{18.6}A_{l14}Cu_{24}$ metallic glass with low lanthanide content. The transformation also manifested itself as a change in short- and medium-range orders. Thus, it was proposed that the lanthanide-solute metallic glasses also inherit 4*f* electronic transition from pure lanthanide element upon compression. This discovery provides a new perspective on the polyamorphic transformation in metallic glasses. So far, polyamorphism denotes a structural transition between different amorphous states, similar to the well-known polymorphism in crystalline materials. Pressure-induced polyamorphism in traditional network-forming glassy state materials, such as ice, silica, and silicon, has been reported. Generally, this polyamorphism involves an open packed structure transforming to a more densely packed one, namely, with an increase in atomic coordination under pressure. Metallic glasses, as a new member of the glass family, are distinct from the traditional network-forming glasses since they have nondirectional metallic bonds in nature. Metallic glasses also exhibit pressure-induced polyamorphism (Fig. 1), although they are spatially densely packed and have a maximum coordination number already.

Directional coordination environment is a key factor in *orientational* C_{60}*-based glasses*, revealing polyamorphism[86,87] and advantageous for various quantum applications (e.g., Refs. 88–90). In short-range ordered species considered above, pressure-driven phase transitions between nonmagnetic amorphous phases gained a unified description in terms of heterophase fluctuations in

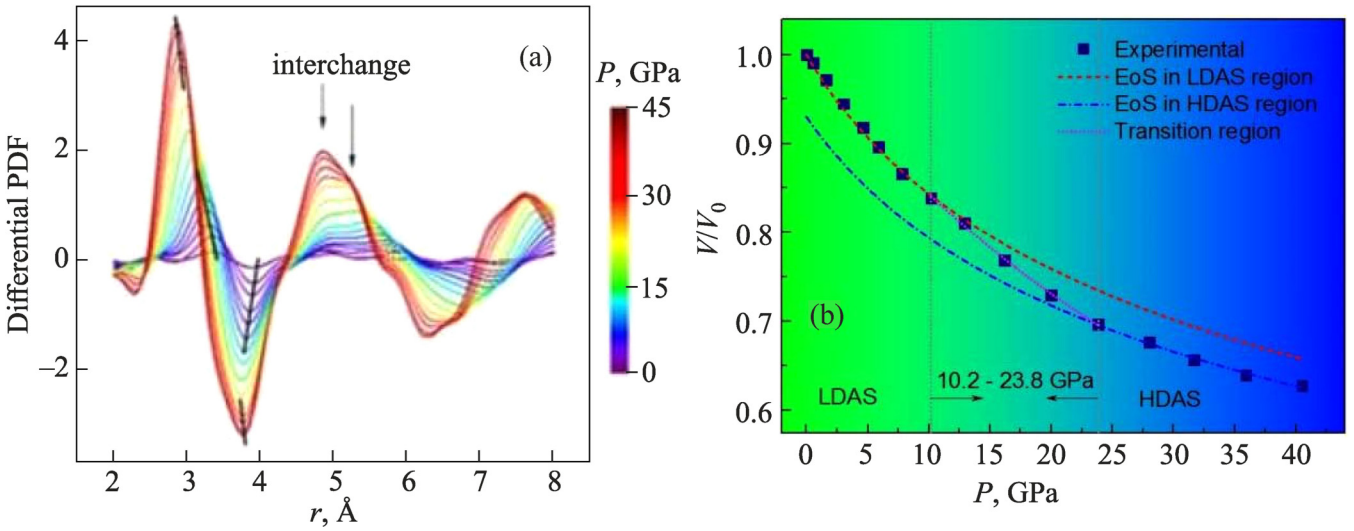

FIG. 1. (a) The typical differential pair distribution functions (PDFs), derived from X-ray powder diffraction data, of the $La_{43.4}Pr_{18.6}Al_{14}Cu_{24}$ metallic glass under different pressures. The black line shows the changes of slope. The arrows show the interchange of status between peak and shoulder,[81] (b) corresponding low-density and high-density states depicted in the phase diagram.[82]

polycluster state[56] and oriented bonds.[91] Moreover, in Ref. 56 resemblance of glass transition in orientation glasses with Ising spin-glass transition is admitted. In fact, more similarities between glass-forming substances and spin species can be traced. The recently found spin-forming phases are the counterparts of water-derived phases, namely, spin-liquid,[92–97] spin-ice,[98,99] and spin-glass[100–111] and references therein).

It would be natural to look for magnetic polyamorphism in addition to electronic,[112] allegedly manifested by vortex states in anisotropic superconductors[113–117] and references therein), "bio",[118,119] macroscopic magnetism under pressure in multilayered graphene oxide.[120] It was reportedly revealed in spin liquid.[97] It is argued that there is discovered the remarkable physics of competing spin-liquid polymorphs in a correlated electron system $FeTe_{1-x}(S, Se)_x$ approaching superconductivity. These results facilitate an understanding of large swaths of recent experimental data in unconventional superconductors. In particular, the phase with lower C2 local symmetry, whose emergence from C4 one, precedes superconductivity, naturally accounts for a propensity for forming electronic nematic states which have been observed experimentally, in cuprate and iron-based superconductors alike. Tracing similarities between structural, electronic, and spin systems, one inevitably arrives at their comparison with quantum phase transitions, mediated by long-range fluctuations and driven by the pressure or magnetic field action.[121,122]

2. SPIN GLASS

Spin glass materials are randomly-frustrated magnetic systems,[98] where the magnetic atoms occupy random positions in lattices, producing random spin frustration, formed by nonmagnetic matrices fixed at the moment of the preparation of the material,[123] considering the quenched disorder. They are a perfect benchmark for understanding the glassy matter and are even called in the Nobel award lecture[101] "the cornucopia for the discovery of the interplay of disorder and fluctuations in physical systems from atomic to planetary scales". A big concern is the glass ordering processes with many observed features, relevant to their non-magnetic counterparts, including their dynamic susceptibilities behavior.[124] As materials that consist of disordered and frustrated magnetic spin alignment with complicated energy landscapes, spin glasses perfectly manifest themselves in a dynamic susceptibility measurement. Their existence was also first confirmed by NMR measurements though. In 1972, sharp cusps were registered in low-frequency ac-susceptibility dependence on the temperature of gold-iron alloy AuFe.[100] These experiments have shown that the temperature at which cusps in ac-susceptibilities measurements occurred is dependent on the iron concentration. As the 1st popular evidence of the existence of a new kind of magnetic material, these experiments attracted attention for a study of this kind of material both experimentally and theoretically for tens of years (for recent developments see Refs. 101–109 and references therein). The origin of the transition to a spin-glass state in zero and finite magnetic field, as well as its thermodynamic stability, were highly debated,[125–128] see also Refs. 101 and 103–106 and references therein. As a result, true phase transition and spin-glass thermodynamic phase existence were proved both theoretically and by experiment.[102]

In terms of the energy landscape, both ferromagnets and antiferromagnets have long-range order ground state or a well-defined global minimum. On the other hand, the frustration of spin glasses introduces complexity into their energy landscapes, as described in Ref. 103. At high temperatures, the system remains in a paramagnetic state, while at temperatures below freezing T_f, there are many metastable states with high energy barriers between them. For the dc-susceptibility measurement after cooling in zero field, all energy valleys are assumed to be similar to each other and the system can be tracked in either one. As heating the sample again, it may not follow the same path to escape from the valley, thus the susceptibility is irreversible. However, after cooling in an applied magnetic field, the energy landscape is biased. Thus, the system can only fall into the valley which has the lowest energy in a magnetic field, with history-dependent waiting times.[130–133] Such patterns of behavior observed first in amorphous metal and insulating Heisenberg spin glasses[107,134,135] and references therein) on the border with Kondo systems, were considered within both Ising and Heisenberg models. It was shown, that in these systems an Ising scenario can be followed in the low field due to the presence of slight anisotropy.[128] The importance of transverse magnetization and chiral glass transition[129] has been considered for such systems[134–137] in line with other slow relaxing systems, like, e.g., spin clusters[138–140] and different types of glasses.[141–143]

3. TRANSVERSE FLUCTUATIONS OF MAGNETIZATION FREEZING

The above-cited results, obtained earlier on Heisenberg spin glasses, are described[127] within a mean-field model of a Heisenberg spin-glass with weak random anisotropy. Recently[108] the spin-glass transition in the strongly frustrated well-known compound Fe_2TiO_5 gained a comprehensive insight. A remarkable feature of this transition, widely discussed in the literature, is its anisotropic properties: the transition manifests itself in the magnetic susceptibly only along one axis, despite Fe^{3+} d^5 spins having no orbital component. It is demonstrated, using neutron scattering, that below the transition temperature $T_g = 55$ K, Fe_2TiO_5 develops nanoscale surfboard-shaped antiferromagnetic regions in which the Fe^{3+} spins are aligned perpendicular to the axis which exhibits freezing. It is shown, that the glass transition may result from the freezing of transverse fluctuations of the magnetization of these regions. A developed mean-field replica theory of such a transition, reveals a type of *magnetic van der Waals effect*. The phase diagram of the infinite-range model of spin-glasses exhibits two mixed phases. In these mixed phases, ferromagnetism and spin-glass order coexist, due to freezing of the transverse degrees of freedom or replica symmetry breaking. For five different Heisenberg spin glass systems, torque experiments were also performed in applied magnetic fields up to 4 T.[135] The Dzyaloshinski–Moriya random anisotropy strengths, the in-field torque onset temperatures, and the torque relaxation were measured. Critical exponents were estimated independently using a standard protocol. The data are strong evidence for a true spin glass ordered state which survives under high applied magnetic fields; they can be interpreted consistently in terms of a chiral ordering model with replica symmetry breaking as proposed in Refs. 104 and 129 and references therein). It is shown,

using Monte Carlo simulation and finite-size scaling analysis, that the Heisenberg spin glass undergoes a finite-temperature phase transition in three dimensions.[136,137] There is a single critical temperature, at which both a spin glass and a chiral glass ordering develop. The Monte Carlo algorithm, adapted from lattice gauge theory simulations, makes it possible to thermalize lattices of size $L = 32$, larger than in any previous spin-glass simulation in three dimensions. High accuracy is reached thanks to the use of the MareNostrum supercomputer. The large range of system sizes studied allows us to consider scaling corrections.[143] In fact, this yields a transition line resembling the so-called GT line of the mean-field model,[127] $|T_{CG}(0) - T_{CG}(H)| \propto H^{1/2}$, although in Ref. 128 it is argued, that the origin of the exponent 1/2 is entirely different due to involvement of transition into the chiral-glass state.

Below, we will focus our effort on a more rarely investigated spin-glass transformation in a common crystal,[144] in contrast to an amorphous, structure environment.[145]

4. SPIN-GLASS POLYAMORPFISM

The experiment was performed on cation-deficient $LaMnO_{3+x}$ single crystal. The well-characterized object was chosen for investigation, because of its earlier ample study by numerous techniques,[146–154] which allowed one to establish reliably its low-temperature magnetic state[150,151] as a frustrated[151] with ferromagnetic spin-glass ordering,[150] at a background, schematically illustrated by Fig. 2 for spin-glass with competing exchange interactions.

In this section, experimental evidence of a spin-glass polyamorphism is presented for a single-crystalline anion-access manganite oxide $LaMnO_{3+x}$ in the vicinity of magnetic ordering transition, induced by a magnetic field. Samples of $LaMnO_{3+\delta}$ with $0 \le \delta \le 0.18$ single crystals (Fig. 3) have been prepared by means of the Chohralsky technique followed by aging at different temperatures and oxygen partial pressures.

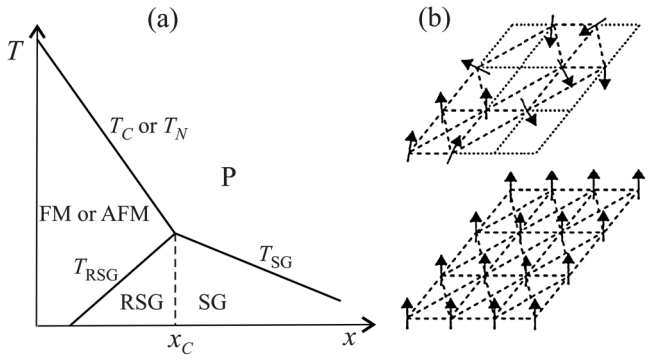

FIG. 2. Spin-glass ordering: (a) Typical phase diagram of a reentrant spin-glass magnetic system showing paramagnetic (P), ferromagnetic (FM), or antiferromagnetic (AFM), spin-glass (SG), and reentrant spin-glass (RSG) phases; x is the concentration of nonmagnetic atoms and (b) random frustration in the presence of competing exchange interactions. Our measurements show the existence of transition between such states, driven by the magnetic field, irreversibility line, slow relaxation, and heat capacity measurements.

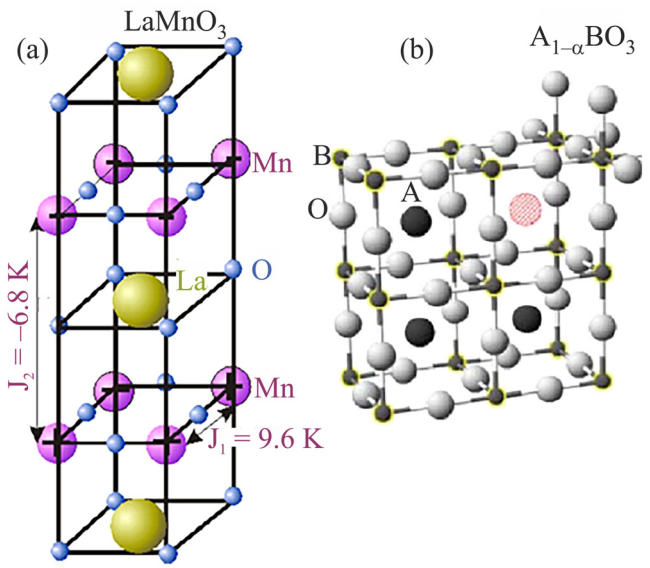

FIG. 3. (a) Competing exchange in $LaMnO_3$; (b) vacancy in cation deficient $LaMnO_3$. The O sites are depicted by large silver spheres, the B sites by small gray spheres highlighted in yellow, and the A sites by large dark spheres. A large hashed red sphere shows an A-site vacancy. By removing oxygen from the A site-deficient unit cell through reduction, some B sites are locally isolated from the parent perovskite into an incipient BO n exsolution (depicted by the red group of atoms in the right panel).

Oxidation is accommodated[149,150] by the formation of cation vacancies to produce $La_{1-\varepsilon}Mn_{1-\varepsilon}O_3$ with $\varepsilon = \delta/(3+\delta)$. Room temperature X-ray diffraction reveals a two-phase region separating an O′-orthorhombic phase stable over $0 \le \delta \le 0.06$ and a rhombohedral phase, stable in the range $0.10 \le \delta \le 0.18$, that transforms below room temperature to an O′-orthorhombic phase. Transport and magnetic studies indicate the following evolution of electronic properties with increasing δ. Oxidation creates small-polaron holes that become increasingly trapped at cation vacancies with decreasing temperatures in the paramagnettic domain. Some of these trapped holes are released on cooling through the onset of long-range magnetic order. In the O′-orthorhombic structure, the trapped holes form superparamagnetic clusters below room temperature that become *magnetically coupled with the onset of antiferromagnetic order in the hole-poor matrix to form a magnetic glass.* The O′-orthorhombic structure sustains the cooperative Jahn–Teller deformation. The rhombohedral phase suppresses the cooperative Jahn–Teller deformation, and the hole-poor matrix becomes ferromagnetic. With increasing δ, the perovskite tolerance factor increases, and at a critical value $t_c \approx 0.97$, a transition from small-polaron to a peculiar nondispersive delocalized state of the conduction electrons occurs below T_c. At the highest δ, these conduction electrons introduce a double-exchange spin–spin coupling in the matrix that varies as $\cos(\theta_{ij}/2)$; this ferromagnetic coupling competes with the antiferromagnetic Mn: t^3–O: $2p_\pi$–Mn:t^3 superexchange, which varies as $\cos\theta_{ij}$. Consequently, an equilibrium θ_{ij} increases with δ to give a cant angle $0° \le \theta \le 180°$, which introduces

metamagnetic behavior of the matrix between clusters and a ferromagnetic magnetization that decreases relatively sharply with increasing δ in the range $0.13 \leq \delta \leq 0.18$. In this way, all conditions for observation of polyamorphism are present, including quench disorder (vacancies), competing interactions (Mn^{3+} anti- and Mn^{4+} ferromagnetic exchange), directional bonds, bridged by octahedral oxygen, and Jahn–Teller ordering of Mn^{3+} orbitals.

Two types of magnetic measurements were performed, which allow to (1) distinguish the magnetic state evolution with temperature and magnetic field by magnetization reversal measurements with field (FC) and zero-field cooling (ZFC)[155] and references therein) in a direct-current (dc) induced magnetic field H_{dc}, and (2) magnetic relaxation times by complex magnetic susceptibility measurements in an alternate-current (ac) induced magnetic field H_{ac}.[132,133,156,157] Thus, in Fig. 4, the H–T diagram is presented, which is built from type 1 measurements by a routine procedure (see, e.g., Ref. 158) of detecting the splitting temperatures of FC and ZFC curves together with dynamic susceptibility and heat capacity measurements. Relaxation time analysis, based on dynamic susceptibility measurements reveals 2 relaxation regimes with H_{dc} variation. For a comparison the calculated[159] phase diagram is presented [Fig. 4(b)]. In Fig. 5 the obtained results are presented in 3D version.

5. RESUME

In addition to previous measurements on metal and insulating spin glasses with predominantly amorphous atomic structure environment, which proved a true phase transition to the spin-glass

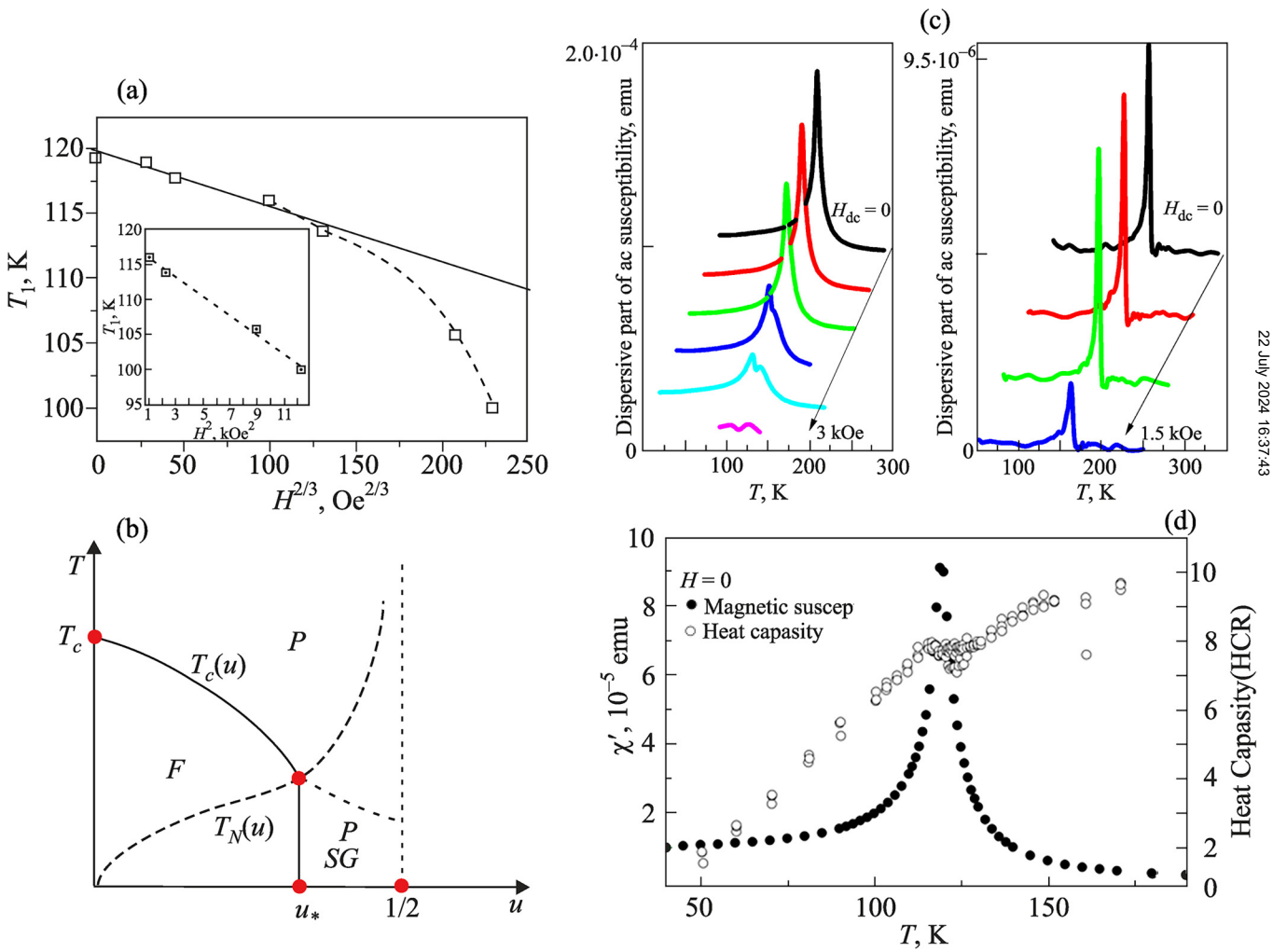

FIG. 4 (a) Dependence T_f ($H^{2/3}$) ($f = 884$ Hz) below the zero-field transition temperature which is deviated from linearity in field strength of $H_T = 1.17$ kOe. Insert shows a square dependence above the field of transition H_T. These results evidence the field-induced transition of the spin-glass state from Ising to a Gabay–Toulouse. (b) For a comparison: Schematic phase diagram of the Ising ferromagnet diluted by antiferromagnetic spin-spin couplings; $T_N(u)$ is the Nishimori line; F, P, and SG denote the ferromagnetic, paramagnetic phase and spin glass, respectively[55] (c and d) The ac- and heat capacity measurements are presented for comparison.

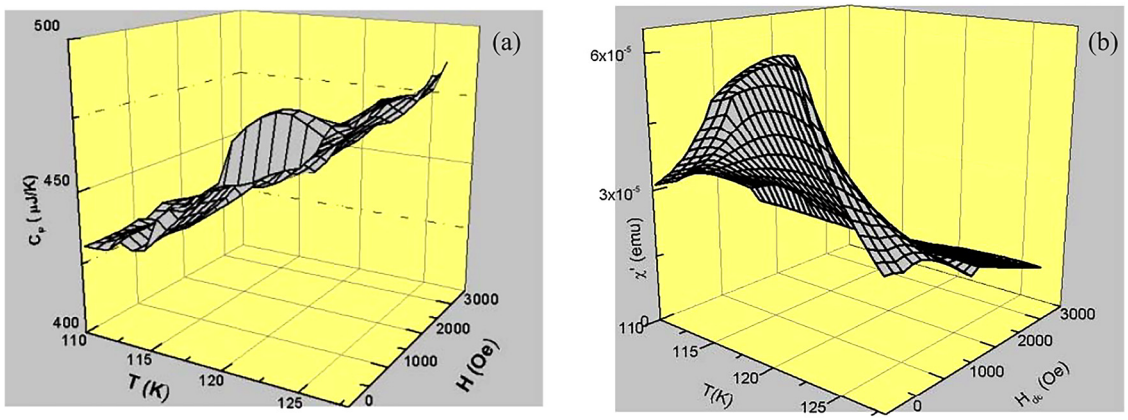

FIG. 5. Heat-capacity (left) and magnetic susceptibility (right) measurements at the transition between spin-glass states.

phase, we present an H–T diagram in line with heat capacity measurements, performed on a cation-deficient $LaMnO_{3+x}$ single crystal with competing exchange interactions, as an evidence of a phase transition between spin-glass states, driven by a magnetic field. This behavior is similar to that, observed in other short-range ordered species, like liquids and nonmagnetic glasses under pressure, which should be described in terms of polyamorfism. All of these phase transitions are characterized by a slow relaxation to equilibrium state, with signatures of hidden order, reminiscent of quantum phase transitions.

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