Letter

Spin and charge density waves in quasi-one-dimensional KMn_6Bi_5

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The recent observation that pressure could suppress antiferromagnetic (AFM) order in quasi-one-dimensional AMn_6Bi_5 Mn-cluster chain materials (A = Na, K, Rb, and Cs) and lead to a superconducting dome offers an alternative Mn-based class of materials with which to study unconventional superconductivity. Using neutron diffraction, we elucidate the exact nature of the previously unknown AFM ground state of KMn_6Bi_5 and report finding transverse incommensurate spin density waves (SDWs) for the Mn atoms with a propagating direction along the chains. The SDWs have distinct refined amplitudes of $\sim$2.46 μ_B for the Mn atoms in the pentagons and $\sim$0.29 μ_B with a large standard deviation for Mn atoms at the center between the pentagons. AFM coupling dominates both the nearest-neighbor Mn-Mn interactions within the pentagon and next-nearest-neighbor Mn-Mn interactions out of the pentagon (along the propagating wave). The SDWs exhibit both local and itinerant characteristics potentially due to cooperative interactions between local magnetic exchange and conduction electrons. Single crystal x-ray diffraction below the AFM transition revealed satellite peaks originating from charge density waves along the chain direction with a **q** vector twice as large as that of the SDW, pointing to a strong real space coupling between them. Additionally, we report a significant magnetoelastic effect during the AFM transition, especially along the chain direction, observed in temperature-dependent x-ray powder diffraction. Our work not only reveals fascinating intertwined spin, charge, and lattice orders in one-dimensional KMn_6Bi_5, but also provides an essential piece of information on its magnetic structure to understand the mechanism of superconductivity in this Mn-based family.

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I. INTRODUCTION

In contrast to conventional s-wave electron Cooper pairing predicted by the Bardeen-Cooper-Schrieffer superconductivity theory [1], unconventional superconductors have revealed nontrivial electronic pairing mediated by suppression of the electronic instabilities of a candidate material via chemical substitution or by the application of external pressure [2]. The underlying mechanisms responsible for unconventional superconductivity remain enigmatic despite numerous groundbreaking discoveries of superconducting families like the famous cuprates [3], iron pnictides [4], heavy fermions [5], and organic Bechgaard salts [6].

Unconventional superconductivity has often been associated with various electronic instabilities such as charge or spin density waves (CDWs or SDWs), with the competing orders providing a fascinating opportunity to combine widely distinct physical phenomena and create new electronic landscapes. The original observation of a static stripe phase in $La_{1.6-x}Nd_{0.4}Sr_xCu\,O_4$ [7], in which coexisting SDWs and CDWs were suggested to correlate with the anomalous suppression of superconductivity, opened the door to extensive experimental and theoretical research that ultimately established the dependence of hole-doped cuprates' superconductivity on intertwined symmetry-breaking long-range or short-range electronic and magnetic correlations [8–27]. Curiously and to the best of our knowledge, coexistence of the SDW and CDW orders has not been observed in other superconducting families, thus, highlighting the urgent need for new materials in which both orders are present and leading to superconductivity.

Superconductivity was recently reported in a series of Mn-based quasi-one-dimensional (Q1D) AMn_6Bi_5 (A = K, Rb, and Cs) materials [28–30] via the suppression of their AFM orders under 9–15 GPa external pressures [30–32]. AMn_6Bi_5 is a model system that offers a rare opportunity for investigations of electronic instabilities in a nuclear structure which

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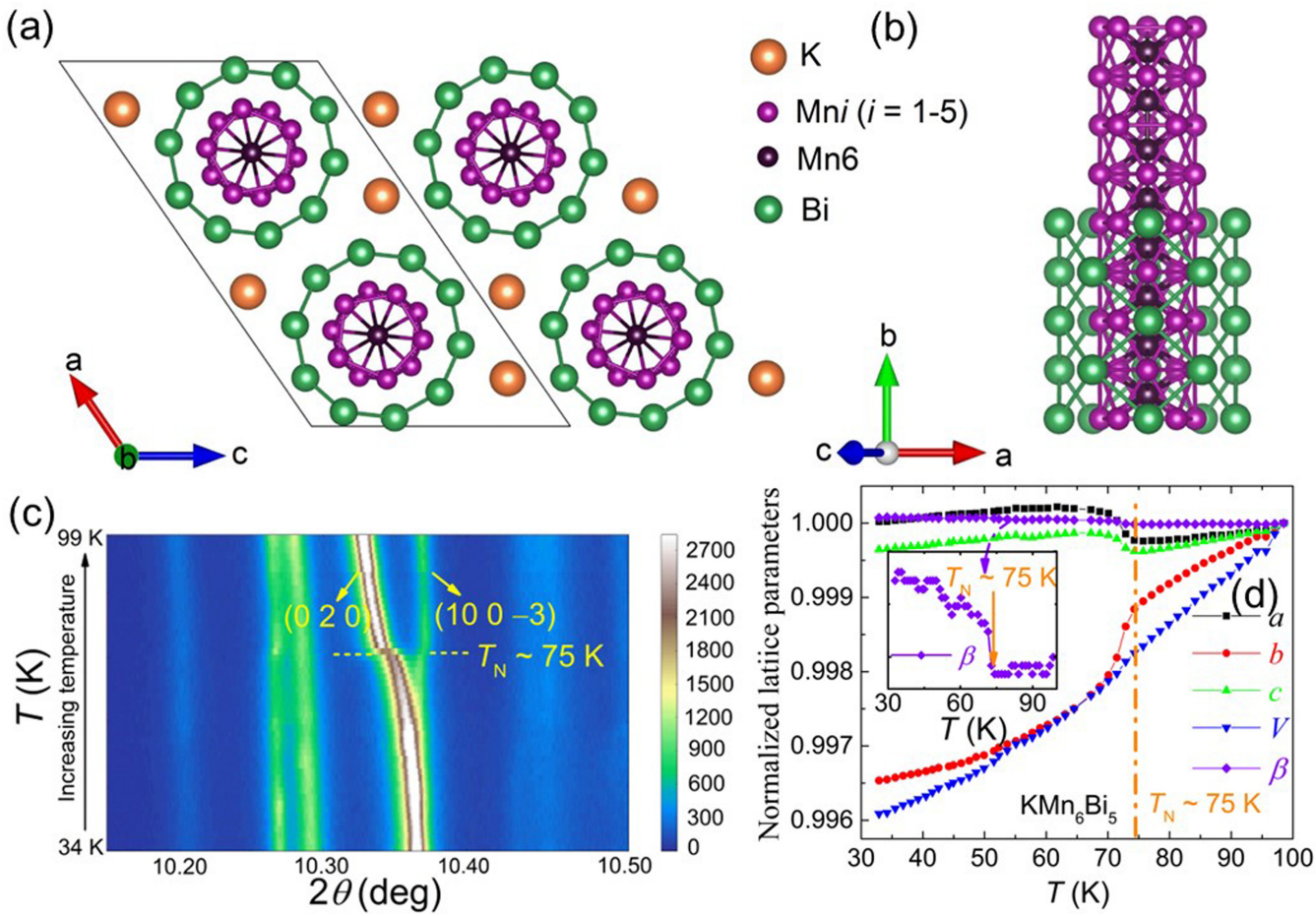

FIG. 1. (a) Top view of the crystal structure of KMn_6Bi_5 down the *b* axis. The bonds were added to help present the nanowire structure of $[Mn_6Bi_5]^-$ more clearly. (b) The structural configuration of the Bi nanotube and the Mn cluster chain in the $[Mn_6Bi_5]^-$ nanowire along the *b*-axis direction. (c) Temperature-dependent powder x-ray diffraction of KMn_6Bi_5 in a selected range of diffraction angles. (d) Temperature-dependent normalized lattice parameters of KMn_6Bi_5. The inset is a magnified view around the phase transition of the monoclinic angle β. The orange dash dot line is a guide to the eye.

consists of unique $[Mn_6Bi_5]^-$ nanowires with counterions *A* intercalated in between to form a Q1D crystal structure [28,29,33,34], see Figs. 1(a) and 1(b). Considering the similar phase diagrams of unconventional superconductivity tuned by high pressure [2,35–37], it is reasonable to classify AMn_6Bi_5 as another class of unconventional superconductors whose underlying physics must be uncovered. Moreover, Q1D systems in which electron-electron correlations and quantum fluctuations become enhanced due to the quantum confinement of electrons [38] are expected to enrich the physics of this superconducting family through various mechanisms. For example, collective electronic instabilities in Q1D systems gave rise to spin-charge separated Tomonaga-Luttinger liquids [39], CDW [40,41], SDW [42,43], and unconventional superconductivity with spin-triplet pairing [44–49].

The magnetic ground state of this emerging family of AMn_6Bi_5 superconductors has remained until now unknown, thus, significantly hindering the ability to properly consider potential pairing interactions and to make meaningful comparisons with the broader family of unconventional superconductors. In the present work, we use neutron single crystal diffraction to solve this conundrum, demonstrating the formation of multiple AFM amplitude-modulated SDWs in KMn_6Bi_5. Moreover, using x-ray single crystal diffraction, we discovered a CDW with a propagation vector twice as large as that of the SDW. The observation of CDWs in coexistence with SDWs as a prelude to superconductivity in KMn_6Bi_5 is of paramount importance since it offers a system to the condensed matter community second only to the hole-doped cuprate superconductors. Our discovery will enable further studies to elucidate the nature of the intertwined orders in diverse AMn_6Bi_5 solid solutions where *A* is tuned by various mixtures of the alkali metals.

II. EXPERIMENTAL DETAILS

The preparation methods of samples for neutron and x-ray diffraction are described in the Supplemental Material (SM) [50]. Synchrotron powder and single crystal x-ray diffraction were performed at beamlines 11-BM-B and 6-ID-D of the Advanced Photon Source at Argonne National Laboratory (ANL), respectively. Magnetic structure determination and order parameter scans were performed using neutron powder diffraction at beamline HB-2A and neutron single crystal diffraction at beamline HB-3A [51] at the high flux isotope reactor (HFIR) at Oak Ridge National Laboratory (ORNL).

Data collection details, processing, and analysis are provided in the SM [50]. Crystal and magnetic structures were plotted using the software VESTA [52].

III. RESULTS AND DISCUSSION

Structural properties. KMn_6Bi_5 crystallizes in a monoclinic crystal structure (no. 12, $C2/m$) consisting of two $[Mn_6Bi_5]^-$ in one unit cell [28]; see Fig. 1(a). Each $[Mn_6Bi_5]^-$ nanowire can be decomposed into a chain of corner-shared Mn-centered icosahedral clusters (13 Mn atoms) encapsulated inside a Bi nanotube; see Fig. 1(b). The Mn clusters consist of five independent Mn sites (Mn1–Mn5) that form two flat pentagons lying within the ac planes, at y coordinates of 0 and 0.5, with an additional independent Mn6 site (referred to as central Mn) occupying central positions, at y coordinates of 0.25 and 0.75, between any two adjacent pentagons stacked along the b axis. The Mn pentagons are structurally related via the symmetry operators of the monoclinic $C2/m$ space group. No sign of any structural symmetry transformation was detected across its AFM transition from temperature-dependent x-ray powder diffraction measurements. The temperature-dependent behavior of the diffraction data agrees with an overall positive thermal expansion of the material and the anomalies at ∼75 K [the opposite responses of the peaks (10 0 −3) and (0 2 0) during the transition] imply significant anisotropic magnetoelastic coupling between its crystal lattice and magnetic order [53,54]; Fig. 1(c). Additional peaks are tracked as shown in Fig. S1 of the SM [50].

Quantitative temperature-dependent lattice parameters of KMn_6Bi_5 determined from Rietveld refinements are shown in Fig. 1(d). With the exception of a subtle slope change of the unit cell volume, all the lattice parameters display a clear anomaly at ∼75 K. It is worth emphasizing the rapidly decreasing lattice parameter b after the system enters the AFM state, which is ascribed to strong AFM exchange magnetostriction [55] within the Mn cluster chains as described below. In contrast, the a and c lattice parameters both exhibit a smaller increase just below the paramagnetic to antiferromagnetic phase transition.

Given that the interchain magnetic interactions are much weaker than their intrachain counterparts, one can argue that the negative thermal expansion displayed by a and c results from elastic strains produced by compression of the lattice along the chain direction (i.e., the b axis). On the other hand, the relatively larger positive thermal expansion along the b axis, Fig. 1(d), suggests that one-dimensional magnetic interactions along the chain direction play a dominant role in determining the overall magnetoelastic properties of the material below 99 K.

Incommensurate spin density waves (ISDWs). To determine the magnetic structure of KMn_6Bi_5, single crystal neutron diffraction was performed. The high quality of our single crystal is demonstrated by the excellent goodness-of-fit and agreement factors obtained at 4 K for the refined nuclear structure as shown in Fig. S2(a) and Table S1 in the SM [50]. Reciprocal space scans revealed the existence of a set of reflections of magnetic origin at temperatures below 75 K, which we successfully indexed using an incommensurate propagation vector $\mathbf{q_s} = (1, 0.418, 0)$. This result is markedly different from any of the theoretical commensurate magnetic models proposed by Chen *et al.* [29] and likely indicates a complex energy landscape with numerous competing magnetic interactions. An order parameter scan was performed using the magnetic reflection (1 0.418 1) and gave a fitted critical component β of 0.15(3) for KMn_6Bi_5 [Fig. S2(c) in the SM [50]], typical of systems with reduced dimensionality and much smaller than the theoretical value of 0.37 for three-dimensional Heisenberg systems [56], which agrees well with the proposed Q1D magnetic interactions [57,58].

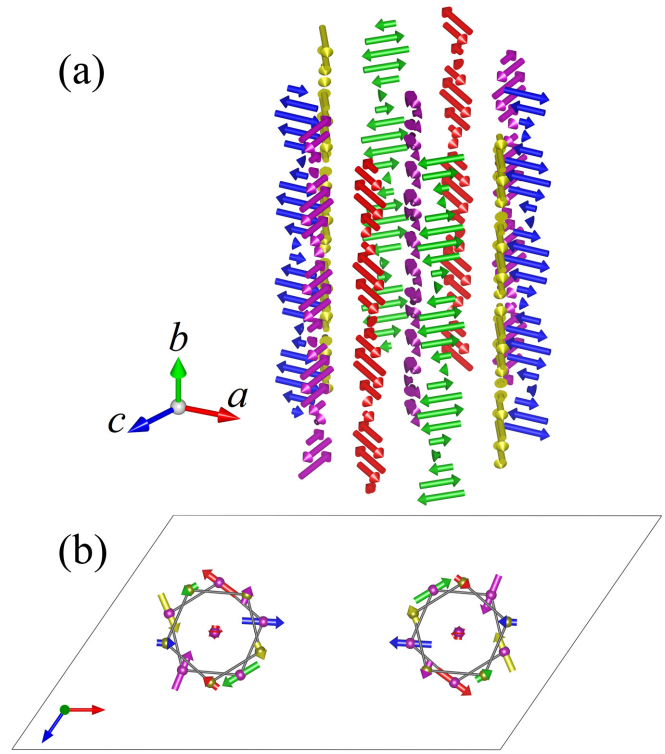

FIG. 2. (a) Incommensurate spin density wave (ISDW) magnetic structure of KMn_6Bi_5. The structure is plotted over 25-unit cells stacked along the b axis. The unit cell's b parameter is significantly compressed to help visualize the waves over many unit cells. Waves passing through both Mn pentagons are shown together with the chain Mn (purple) through the pentagons' axis. (b) A [010] view of the base unit cell showing the two Mn pentagons (purple spheres at $y = 0$; dark yellow spheres at $y = 0.5$). Arrows of the same color are located on two Mn sites that are symmetry related by C centering. Detailed views are shown in Fig. 3. See text for more details.

Given the incommensurate $\mathbf{q}$ vector hosted by a monoclinic structure in which six independent Mn sites describe a total of 24 Mn ions per unit cell, one would expect the resulting magnetic structure to be of low symmetry. To start, we considered the highest symmetry magnetic models, each of which had the same six symmetry distinct Mn sites as the nuclear structure, however, in our modeling we found it necessary to go beyond these structures and further break the C-centering symmetry to obtain reasonable agreement with the observed intensities. This symmetry breaking increased the number of independent Mn sites to 12 per unit cell; see Figs. 2 and 3 and magnetic modeling in the SM [50]. In our final model with an agreement

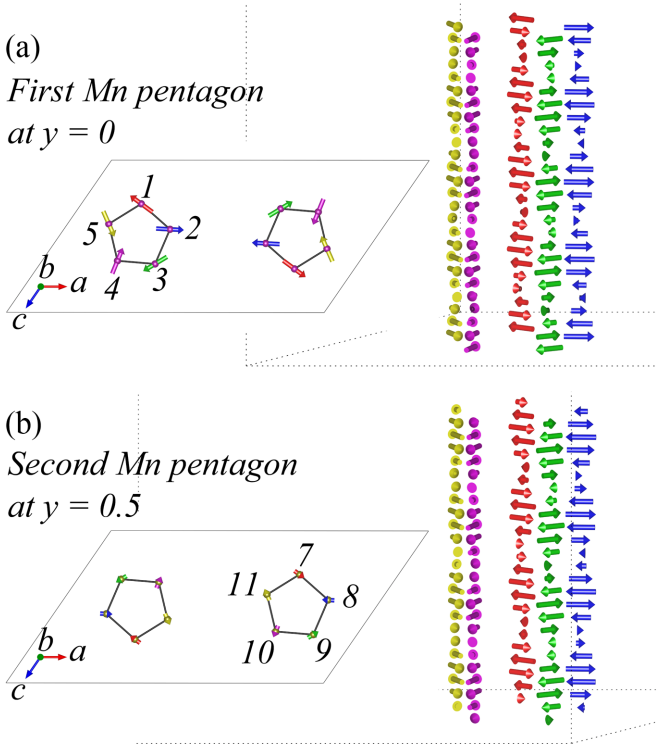

FIG. 3. ISDWs passing through (a) the first Mn pentagon located at $y = 0$ as shown to the left of the figure and (b) the second pentagon located at $y = 0.5$. Notice the AFM coupling along the wave propagation direction and that the sublattices are out of phase (see text for more details). Numbers within the panels refer to Mn1 through Mn5 (a) and Mn7 through Mn11 (b).

factor of about 8% [Fig. S2(b)], amplitude modulated SDWs with a period of about 12-unit cells (∼55.4 Å) along the b axis are observed. The twofold rotation symmetry along the (0.5, y, 0.5) axis transforms the magnetic moments (u, 0, w) of Mn atoms in one Mn-cluster chain to ($-u$, 0, $-w$) of Mn atoms in the other Mn-cluster chain as shown in Fig. 3. Each one Mn-atom chain (with a Mn-Mn distance equal to that of the lattice parameter of b) has an independent SDW. For the central Mn sites (Mn6 and Mn12 with $b/2$ Mn-Mn distance), two separate waves are superimposed. We note that the waves passing through the basal pentagons (i.e., at $y = 0$) are ahead of the waves that propagate through the Mn pentagons at $y = 0.5$ by about 284°. This phase shift determined by the strong coupling of SDWs between two neighboring Mn pentagons within the same Mn-cluster chain is likely the driving force leading to breaking the C centering of the parent space group. Other models such as spiral helical magnetic ones were tried, however, they either inadequately modeled the data or else converged to the current solution; see refinement details in the SM [50]. Our model produces an excellent fit to the magnetic intensities observed by neutron powder diffraction data collected at 3 K (Fig. S3).

The above-described structure has 12 independent SDWs on the Mn atoms coupled and propagated together along one single Mn-cluster chain in KMn_6Bi_5. The magnetic configuration can be described by the general formula [59]

$$\mathbf{m_{ji}} = \mathbf{m_i} \cos(-2\pi \mathbf{q_s} \cdot \mathbf{R_j} + \varphi_i) \quad (i = 1{-}24),$$

where $\mathbf{m_{ji}}$ and $\mathbf{m_i}$ with a vector form of (u, 0, w) are the magnetic moments in the jth unit cell and wave amplitudes for the Mni atom, respectively, $\mathbf{R_j}$ is the translation vector, $\mathbf{q_s}$ is the propagation vector, and φ_i is the phase for one certain Mn atom. The magnetic moments and directions of Mn6 and Mn12 cannot be determined accurately from our current data set due to the majority of the magnetic scattering coming from the Mn pentagons which exhibit significantly larger magnetic moments. For clarity, the waves from different Mn pentagons are separated as shown in Figs. 3(a) and 3(b). The refined magnetic structure parameters are presented in Table S2 in the SM [50]. It is important to note that in our model the Mn magnetic moments oscillate along the chain direction, in stark contrast to the predicted helical magnetic model by Chen *et al.* [29] where they are constant along the chain direction, but their directions vary.

All the SDWs along the chain are transverse in KMn_6Bi_5 since the propagating direction is perpendicular to the spin direction. In a conventional transverse SDW, the modulated magnetic moments point in one direction for half of the wave then the reverse direction in the other half as in Cr metal [60], for example. In this work, the component of the vector $\mathbf{q_s}$ along the chain direction is 0.418 which corresponds to a phase shift of 150 ° when moving one unit cell along the chain direction. As a result, the magnetic moments are not only modulated in magnitude as expected for a transverse SDW but also coupled antiferromagnetically between neighboring Mn sites along the wave direction—an unusual configuration which will be discussed later.

SDW instabilities can arise in a low-dimensional metallic system due to electron-electron interactions [61], which is consistent with the metallic 3d transition metal Q1D KMn_6Bi_5. Indeed, carrier concentration decreases during the AFM transition probably ascribed to partial gapping of the Fermi surface [28]. The determination of Fermi surface in KMn_6Bi_5 will be essential to probe if Fermi surface nesting is the major driving force triggering its SDW instability and consequently to understand its impact on superconductivity. A simple calculation of the oxidation state reveals that a total of +14 charges must be accounted for by the six Mn ions assuming K^+ and Bi^{3-} [62]. Our refinements of ∼2.46 μ_B and 0.29 μ_B as the wave magnitudes and their corresponding root mean square values of 1.73 μ_B and 0.21 μ_B for the pentagon and central Mn atoms, respectively, suggest possible charge disproportionation and ordering of the different magnetic moments with five Mn^{2+} (5 μ_B for high spin) making the pentagons and one Mn^{4+} (1 μ_B for low spin) along the pentagon axes. The reduced magnetic moment of Mn in KMn_6Bi_5 can be realized by delocalization of the 3d electrons from Mn-Mn metallic bonding within the chain [28]. The significantly smaller magnetic moment in the central Mn ions is supported by the very short Mn-Mn distance (∼2.28 Å at 4 K) where strong metallic bonding between Mn atoms exists [63]. First-principles calculations on AMn_6Bi_5 do reveal that Mn-3d electrons dominate the density of states around the Fermi level, proving the itineracy of the 3d electrons [29,33]. The small magnetic moments obtained in the model are also nominally consistent with the small effective magnetic moment per Mn atom obtained from the Curie-Weiss fitting [28,31]. The disproportionation of magnetic moments

of Mn atoms in KMn_6Bi_5 is reminiscent of the coexistence of magnetic (2-3 μ_B) and almost nonmagnetic (0.2-0.6 μ_B) Mn atoms in the allotrope α-Mn [64]. Our results agree well with DFT calculations performed by Chen *et al.* [29] from which the central Mn ions are found to have a much smaller magnetic moment than the pentagon Mn. The small magnetic moments in KMn_6Bi_5 may be key to achieving superconductivity by high pressures like CrAs [35] and MnP [36] where fragile AFM can be easily quenched.

While the small effective magnetic moments from central Mn atoms (0.21 μ_B) are similar to those of a typical SDW system [3,6,65], the effective magnetic moments (1.73 μ_B) of Mn in the pentagon in KMn_6Bi_5 are relatively large, indicating that magnetism comes from both localized and itinerant electrons as in GdSi, for example, where local magnetic moments and itinerant electrons form a cooperative magnetic configuration [66]. In terms of a local magnetic picture, the Mn-Bi-Mn magnetic exchange geometry in KMn_6Bi_5 is similar to that in the prototypical insulator $BaMn_2Bi_2$ where AFM coupling dominates both nearest and next-nearest neighboring Mn-Mn interactions [67]. The canted AFM configuration for two nearest-neighbor Mn atoms in the pentagon in KMn_6Bi_5 can therefore be explained as a compromise arising from geometric frustration and AFM exchange coupling via Bi atoms in the same plane; see Fig. 2. AFM coupling in the waves for Mn pentagons can be understood by next-nearest-neighbor Mn-Mn magnetic exchange using Bi atoms sandwiched between them as a bridge [29]. In contrast to other pressure-induced superconductors containing 3*d* electrons such as CrAs [35] and MnP [36] with some degree of ferromagnetic interactions, AFM dominates in KMn_6Bi_5 with a one-dimensional and strongly coupled magnetic sublattice, which poses a constraint on creating a theoretical model to understand its superconductivity under high pressure.

Incommensurate charge density waves (ICDWs). In addition to single crystal neutron diffraction, we performed single crystal synchrotron x-ray diffraction which surprisingly revealed the existence of satellite reflections, in addition to the main Bragg peaks below the magnetic phase transition (at 30 K), as shown in Fig. 4. These extra peaks can be successfully indexed using a propagation vector $\mathbf{q_c} \approx (0,\ 0.83,\ 0)$, suggesting the existence of a CDW along the chain direction. The charge propagation vector $\mathbf{q_c}$ is almost double that of the incommensurate modulated component of the SDW vector $\mathbf{q_s}$ indicating their strong coupling in real space as is also observed for example in Cr [60,68], underdoped cuprates [69], and nickelates [70]. *C*-centering symmetry in the crystal structure is still preserved at 30 K based on the rule of the extinction condition for the main reflections despite it being broken by the magnetic structure. Integrated synchrotron intensities at 30 and 297 K are shown in Figs. S4 and S5 in the SM [50].

Compared to the SDW, the CDW is more subtle in KMn_6Bi_5, pointing to a second-order-effect consequence of SDWs. No additional anomalies related to the CDW order can be identified in resistance, magnetic susceptibility, or heat capacity measurements. One possible scenario may relate the CDW to magnetostriction effects between neighboring magnetic moments which result in a charge modulation period equal to half of the spin modulation period in the real space

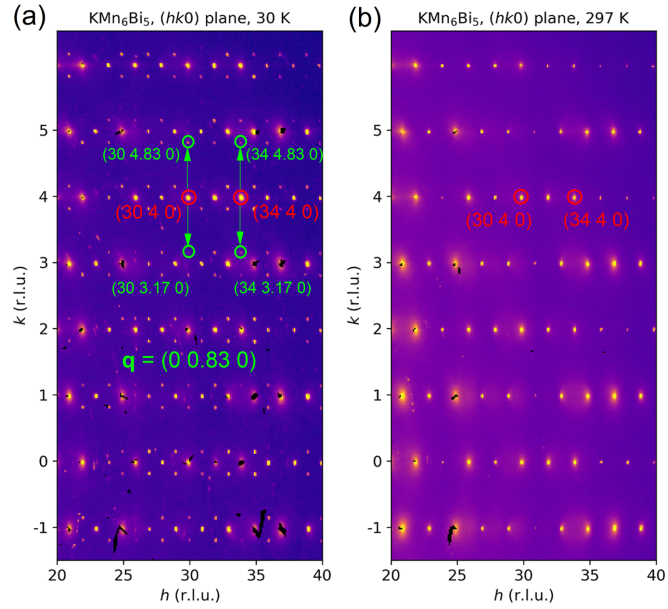

FIG. 4. Integrated synchrotron x-ray diffraction intensities measured in the (*hk*0) plane for KMn_6Bi_5 at (a) 30 K and (b) 297 K, respectively. Examples of CDW satellite peaks and their corresponding **q** vector are shown.

since it does not depend on the directions of those neighboring moments [71]. An incommensurate superspace-group structure refinement is essential to resolve the detailed charge modulation, which, however, cannot be carried out due to the limited quality of the current data set.

IV. CONCLUSIONS

Using a combination of neutron and synchrotron x-ray scattering, we successfully determined the coexistence of complex incommensurate charge and spin density waves in KMn_6Bi_5. Out-of-phase incommensurate SDWs with sizeable magnitudes of about 2.46 μ_B propagate through the Mn pentagons along the *b*-axis direction. In contrast, waves with a small magnitude of no more than 0.29 μ_B pass through the central Mn chain. The magnetism in KMn_6Bi_5 has the dichotomy of both local and itinerant characteristics. AFM interactions dominate both within the Mn pentagons and out of the pentagons along the waves in KMn_6Bi_5. The Mn-cluster chains are antiferromagnetically and ferromagnetically coupled along the *a* and *c* axis, respectively. The reduced magnetic moments of Mn in KMn_6Bi_5 are believed to be an important ingredient to quench the fragile AFM order and achieve superconductivity under high pressure. The CDW found in this work is much subtler than the SDW and probably originates from a magnetostriction effect of the SDW indicating a strong coupling between the two instabilities [71]. KMn_6Bi_5, as a unique Q1D system, demonstrates strong interactions among different degrees of freedom such as spin, charge, and lattice. The intertwined orders, including SDW, its induced CDW at ambient pressure, and superconductivity under high pressure in KMn_6Bi_5 provide a fascinating playground to develop microscopic theories to explain the origins of these quantum phases and their intimate interplay with the

potential for complicated landscapes as in the superconducting cuprates [72].

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