Elastocaloric signatures of symmetric and antisymmetric strain-tuning of quadrupolar and magnetic phases in DyB$_2$C$_2$

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The adiabatic elastocaloric effect measures the temperature change of a given system with strain and provides a thermodynamic probe of the entropic landscape in the temperature-strain space. Here, we demonstrate that the DC bias strain-dependence of AC elastocaloric effect allows decomposition of the latter into symmetric (rotation-symmetry-preserving) and antisymmetric (rotation-symmetry-breaking) strain channels, using a tetragonal f-electron intermetallic DyB$_2$C$_2$—whose antiferroquadrupolar order breaks local fourfold rotational symmetries while globally remaining tetragonal—as a showcase example. We capture the strain evolution of its quadrupolar and magnetic phase transitions using both singularities in the elastocaloric coefficient and its jumps at the transitions, and the latter we show follows a modified Ehrenfest relation. We find that antisymmetric strain couples to the underlying order parameter in a biquadratic (linear-quadratic) manner in the antiferroquadrupolar (canted antiferromagnetic) phase, which are attributed to a preserved (broken) global tetragonal symmetry, respectively. The broken tetragonal symmetry in the magnetic phase is further evidenced by elastocaloric strain-hysteresis and optical birefringence. Additionally, within the staggered quadrupolar order, the observed elastocaloric response reflects a quadratic increase of entropy with antisymmetric strain, analogous to the role magnetic field plays for Ising antiferromagnetic orders by promoting pseudospin flips. Our results demonstrate AC elastocaloric effect as a compact and incisive thermodynamic probe into the coupling between electronic degrees of freedom and strain in free energy, which holds the potential for investigating and understanding the symmetry of a wide variety of ordered phases in broader classes of quantum materials.

Significance

Characterizing the symmetries of ordered phases is paramount in the study of strongly correlated electron systems, and anisotropic lattice distortion/strain offer a fruitful avenue for probing and tuning these phases. Our experimental framework resolves symmetric (rotation-symmetry-preserving) and antisymmetric (rotation-symmetry-breaking) strain effects in free energy using a thermodynamic quantity, the elastocaloric effect. By tracing out the entropy landscape of an f-electron intermetallic in the temperature-strain plane, we demonstrate that the strain-even (odd) component of the elastocaloric response corresponds to symmetric (antisymmetric) lattice deformation modes distinctively coupled to the underlying electronic degrees of freedom. Along with the derived Ehrenfest relation, the elastocaloric effect has potential applications in placing experimental constraints on the spatial symmetry of wider classes of emergent phases in quantum materials.

The authors declare no competing interest.

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irreducible representations allows the introduction of elastoresponse tensor (15), whose components in distinct symmetry channels enabled distinguishing different microscopic mechanisms of coupling between strain and conduction electrons (16–18). These works demonstrate that decomposing an applied strain into distinct symmetry channels provides an organizing principle toward a systematic understanding of experimentally obtained strain responses. This can be especially important for systems which undergo phase transitions with spatially anisotropic order parameters—examples include nematic, spin/charge density wave, orbital and multipolar orders.

In the present work, adding to the symmetry-resolving capabilities of anisotropic strain, we apply a symmetry-decomposition framework to a strain-based thermodynamic quantity, namely, the adiabatic elastocaloric coefficient. We employ this technique to unravel the interplay between symmetric (rotational–symmetry–preserving) and antisymmetric (rotational–symmetry–breaking) strains and the underlying anisotropic f-electron degrees of freedom in a tetragonal intermetallic DyB₂C₂. The localized f electrons in DyB₂C₂ are known to host an antiferroquadrupolar order at low temperature; here at each Dy site the local tetragonal symmetry is broken with time-reversal symmetry intact (this is characterized by a finite electrical quadrupolar moment), and the global tetragonal symmetry is retained by a staggered pattern of the quadrupolar moments along the c-axis (19). Locally resembling electronic nematicity, a considerable coupling is expected between antisymmetric strains that also breaks tetragonal symmetry with the local quadrupole moments (20), while how such coupling tunes a staggered order has not been experimentally determined. We therefore employ the symmetry resolution of the elastocaloric effect to investigate how the staggered anisotropic order is tuned in both symmetric and antisymmetric strain channels. Numerous other strongly correlated electron systems exhibit similar spatially modulated rotational–symmetry–breaking orders—such as orbital ordering in transition metal oxides (21, 22), spin and charge stripe order in the low temperature tetragonal phase in La₂₋ₓBaₓCuO₄ (23, 24), along with a few f electron-based “hidden order states” (25)—we anticipate the methodology demonstrated in our study to be potentially relevant for such systems.

The article is organized as follows: We first discuss the elastocaloric effect itself and its symmetry properties in Section ; in Sections and , we use the elastocaloric effect to categorize the symmetric and antisymmetric strain effects to the quadrupolar and magnetic phase transitions in the system; we then focus on the antisymmetric strain responses away from the phase transitions in Section and in Section discuss broader scopes of using the symmetry decomposition aspect of the elastocaloric effect as an experimental probe of spatially anisotropic orders and fluctuations.

**Elastocaloric Effect and Its Symmetry Decomposition**

The adiabatic elastocaloric coefficient is defined as the temperature T change of a given system in response to strain $\epsilon$ as $(\partial T/\partial \epsilon)_S$, and it has recently emerged as an incisive tool for the study of strain responses of a number of quantum materials (26, 27). In these studies, the elastocaloric coefficients are experimentally probed through the measurement of a temperature oscillation in response to an AC strain, at a frequency such that a quasi-adiabatic condition is achieved (26). To illustrate the physical origin of the elastocaloric effect, in Fig. 1A we depict with a thick two-headed arrow the thermodynamic trajectory of an AC elastocaloric measurement on a generic system in the $T$–$\epsilon$ phase space. As adiabatic processes are confined along the black solid isentropic contours, the normalized temperature oscillation (adiabatic elastocaloric coefficient) can be related to the $\epsilon$-derivative of entropy $S$ in the isothermal condition (26):

$$\left(\frac{\partial T}{\partial \epsilon}\right)_S = -\frac{T}{C_v} \left(\frac{\partial S}{\partial \epsilon}\right)_T. \quad [1]$$

Here $C_v$ is the heat capacity at fixed $\epsilon$. Eq. 1 implies that the elastocaloric effect can be in turn used to characterize the entropy landscape with $\epsilon$. We note that strictly speaking strain $\epsilon_{ij}$ is a 3 × 3 tensor and in Eq. 1 we use $\epsilon$ to denote the experimentally generated linear combinations of $\epsilon_{ij}$ components; the $\epsilon$-derivatives $(\partial T/\partial \epsilon)_S$ and $(\partial S/\partial \epsilon)_T$ are defined for all other components of $\epsilon_{ij}$ being held to zero. We return to the specific combination of strain tensor components shortly. Unlike transport-based strain derivatives such as elastoresponse, whose interpretation requires an understanding of the conduction electron scattering processes (15), $(\partial T/\partial \epsilon)_S$ provides a direct thermodynamic probe into the coupling between strain and fluctuations.

**Fig. 1.** Adiabatic elastocaloric effect and its symmetry properties. (A) Schematic of adiabatic AC elastocaloric effect in a temperature ($T$)-strain ($\epsilon$) phase space, where the black solid lines are isentropic contours. The intensity of gray shade indicates the value of entropy $S$. (B) Schematic of $\frac{1}{2}(\epsilon_{xx}+\epsilon_{yy})(\epsilon_{A1g})$ (blue box) and $\frac{1}{2}(\epsilon_{xx}-\epsilon_{yy})(\epsilon_{B1g})$ (red box). The gray dashed box indicates an undeformed tetragonal lattice. (C) and (D) Schematic of the lowest-order evolution of $S$ (C) and $dT/\partial \epsilon$ (D) with $\epsilon$ in the respective symmetric $\epsilon_{A1g}$ (blue) and antisymmetric $\epsilon_{B1g}$ (red) strain channels (see text).
underlying degrees of freedom in a given system in free energy (28).

**Elastocaloric Effect in Dy$_2$B$_2$C$_2$: Symmetric and Antisymmetric Strain-Tuning of the Antiferroquadrupolar (AFQ) Phase Transition**

Having outlined the symmetry properties of the elastocaloric effect, we apply the above framework to investigate the strain response of a tetragonal rare earth intermetallic Dy$_2$B$_2$C$_2$. Dy$_2$B$_2$C$_2$ crystallizes in the space group $P4/mnbm$; as shown in Fig. 2A, the Dy square lattice layers are spaced by planar networks of B-C octagon and parallelogram motifs. In Fig. 2B, we show an optical image of the experimental setup, where a piece of Dy$_2$B$_2$C$_2$ single crystal shaped into a long, thin plate is mounted on a strain cell and the stress thus experienced by the sample is uniaxial along the in-plane [100] axis (which we denote as $x$). The temperature oscillation generated by the AC strain via the elastocaloric effect is measured using the thermometer/thermocouple attached to the surface of the sample, and the AC strain frequency is selected such that the system approaches a quasi-adiabatic condition (Materials and Methods and SI Appendix). In this experiment, we control and measure $\varepsilon_{\parallel}$, while $\varepsilon_{\perp}$ and $\varepsilon_{2}$ are left unconstrained; based on Poisson ratios $v_{12} = -d_{\varepsilon_x}/d_{\varepsilon_y} = 0.45$ and $v_{13} = -d_{\varepsilon_y}/d_{\varepsilon_x} = 0.08$ estimated from the elastic moduli tensor of LuB$_2$C$_2$ (32) (Materials and Methods and SI Appendix), we linearly decompose the induced deformation into its components in symmetric and antisymmetric channels with their relative strength as $S = \varepsilon_{\phi} = \frac{1}{2}(\varepsilon_{\phi_{xx}} + \varepsilon_{\phi_{yy}})$ to $0.72 \pm 0.08$ (these numbers are normalized with respect to $\varepsilon_{\phi_{xx}}$). The decomposition is schematically illustrated in Fig. 2C: The experimentally generated deformation is shown as a green cuboid and its symmetric $A_{1g}$ (antisymmetric $B_{1g}$) component as blue (red) cuboids. We report the measured elastocaloric coefficient as the oscillation amplitude of $T$ normalized by that of $\varepsilon_{\phi_{xx}}$ which we experimentally control (therefore the measured quantity can be expressed as $\frac{dT}{d\varepsilon_{\phi_{xx}} (s = \text{const})}$; hereafter we denote this as $dT/d\varepsilon_{\phi_{xx}}$ unless otherwise stated. We note that $dT/d\varepsilon_{\phi_{xx}}$ thus defined contains both $\varepsilon_{\phi_{1g}}$ and $\varepsilon_{\phi_{2g}}$-components (Materials and Methods).

The Dy sites in Dy$_2$B$_2$C$_2$ possess a tetragonal local symmetry and the crystal field ground state of Dy$_5^3$ (4$^2$) is a quarter $J_{z} \approx |\pm \frac{1}{2}, | \pm \frac{3}{2}\rangle$ within the $J = \frac{15}{2}$ manifold (33). Here $J$ is the total angular momentum operator. This $f$ electron quartet provides a basis for a two-step phase transition to fully release the $R$ in $4$ entropy ($R$ is the gas constant) and an avenue to order in a time-reversal-symmetric quadrupolar channel (order parameter characterized by $O_{x_{z} - y_{z}} \equiv J_{x}^{2} - J_{y}^{2}$ and $O_{xy} \equiv J_{x}J_{y} + J_{y}J_{x}$) prior to magnetic order (order parameter characterized by $J_{x}, J_{y}, J_{z}$). In the quadrupolar order that sets in at about 25 K, the charge distribution of the $f$ electrons breaks the high-temperature local fourfold rotational symmetry and the resulting quadrupole moments (linear superposition of $O_{x_{z} - y_{z}}$ and $O_{xy}$) in neighboring layers are orthogonal to each
other, forming an antiferroquadrupole (AQF) state (19, 34). In this AQF state, a combined $C_4$ and translational symmetry (translation operation performed along $c$) is preserved, analogous to the combined time-reversal and translational symmetry of a Néel order. At 16 K an additional phase transition into a noncollinear canted antiferromagnetic (CAFM) phase takes place; there magnetic moments develop within the $ab$-plane and approximately perpendicular to the charge clouds, resulting in a net magnetization along the [100] axes (19, 35). The two-phase transitions can be traced in the heat capacity $C_T$ with strain $\epsilon_{xx}$ (measured using a relaxation method) in Fig. 2D (36). We define the higher and lower $T$ anomalies as $T_Q$ (AFQ) and $T_N$ (CAFM), respectively; both phase transitions have been reported to be of second-order nature (19).

In Fig. 2E, we show $dT/d\epsilon_{xx}$ taken at nominally zero bias strain $\epsilon_{xx} = 0$. We observe two anomalies with opposite signs in the $T$-dependence of $dT/d\epsilon_{xx}$, corresponding to $T_Q$ and $T_N$ in $C_T$, respectively. The qualitative resemblance of the singularities in $dT/d\epsilon_{xx}$ and $C_T$ is related to the fact that both $C_T$ and $dT/d\epsilon_{xx}$ are second-order derivatives of thermodynamic potentials. In analogy to the Ehrenfest relation in thermal expansion at second-order phase transitions (37), we derive a modified Ehrenfest relation for $dT/d\epsilon_{xx}$ (see SI Appendix A):

$$\frac{dT_C}{d\epsilon_{xx}} = \frac{\Delta[C(dT/d\epsilon_{xx})]}{\Delta C},$$

[4]

where the evolution of critical temperature $T_C$ with strain $dT_C/d\epsilon_{xx}$ is related to the jump of $C(dT/d\epsilon_{xx})$ along with that in the heat capacity $C_T$ at the phase transition. From $dT/d\epsilon_{xx}$ shown in Fig. 2E and assuming $C_T$ in Fig. 2D provides a reasonable approximation for $C$ in Eq. 4 given the small compressibility of solids, we estimate $dT_N/d\epsilon_{xx} = 45$ K and $dT_Q/d\epsilon_{xx} = -35$ K near $\epsilon_{xx} = 0$.

Having demonstrated that $dT/d\epsilon_{xx}$ can be used to track both AFQ and CAFM phase transitions in DyB$_2$C$_2$, we proceed to the evolution of $dT/d\epsilon_{xx}$ between 20 K and 30 K at selected $\epsilon_{xx}$ in Fig. 3A. A nonzero $\epsilon_{xx}$ introduces a number of changes, which we outline as follows. First, the location of $T_Q$ (marked by arrows in Fig. 3A) moves with $\epsilon_{xx}$, and the trend is summarized in Fig. 3B. Second, at $T_Q$, a deep, negative jump at the most tensile $\epsilon_{xx}$ gradually evolves into a positive, step-like feature at the most compressive $\epsilon_{xx}$. We note that there are intermediate $\epsilon_{xx}$ values (between $-0.15\%$ and $-0.21\%$) where the singularity associated with $T_Q$ appears vanishingly small; the $\epsilon_{xx}$-evolution of the jump at $T_Q$ of $dT/d\epsilon_{xx}$ (i.e. $\Delta[dT/d\epsilon_{xx}]$, estimated as $dT/d\epsilon_{xx}(T = T_Q) - dT/d\epsilon_{xx}(30 \text{ K})$) is summarized in Fig. 3C. Finally, away from the phase transitions both above and below $T_Q$, we observe a continuous variation of $dT/d\epsilon_{xx}$ with $\epsilon_{xx}$; we return to these behaviors in section 4.

Eq. 4 suggests that given $dT/d\epsilon_{xx}$ traces at various bias $\epsilon_{xx}$, two independent means exist to trace the strain-dependence of a phase transition: either focusing on the singularity in $dT/d\epsilon_{xx}$ marking $T_Q$, or the size of jump in $dT/d\epsilon_{xx}$ in conjunction with $C$ at $T_Q$. The former and latter views of the AFQ phase transition are contrasted in Fig. 3B and Inset of $C$, respectively. In the latter, we assume that $C$ for both the ordered and disordered phases near $T_Q$ are independent of $\epsilon$. In Fig. 3B, $T_Q$ appears to evolve with $\epsilon_{xx}$ both in a linear $\epsilon_{xx}$ and quadratic $\epsilon_{xx}^2$ manner. Alternatively, $\Delta[C(dT/d\epsilon_{xx})]/\Delta C$ shown in Fig. 3C, Inset appears linear with $\epsilon_{xx}$ over the entire $\epsilon_{xx}$ range, and a linear
fit \( A + B \epsilon_{\text{xx}} \) yields \( A = -47.1(7), B = -2.49(4) \times 10^4 \). Following Eq. 4, \( \Delta(C(dT/d\epsilon_{\text{xx}}))/\Delta C \) should reflect the \( \epsilon_{\text{xx}} \)-evolution of \( dT_Q/d\epsilon_{\text{xx}} \); in Fig. 3B we compare an integration of \( A + B \epsilon_{\text{xx}} \) multiplied by a factor to account for the imperfect adiabaticity in our experiments (black solid curve) with the directly traced \( T_Q(\epsilon_{\text{xx}}) \) (blue symbols).\(^1\) That the integrated curve agrees with the bare \( \epsilon_{\text{xx}} \)-dependence of \( T_Q \) provides an experimental demonstration of the validity of the modified Ehrenfest relation Eq. 4 and additionally suggests that our assumption that the heat capacity is not strongly affected by \( \epsilon \) is valid. We note that a direct polynomial fit to \( T_Q(\epsilon_{\text{xx}}) \) yields uncertainties of the \( \epsilon_{\text{xx}} \)(\( \epsilon_{\text{xx}}^2 \))-coefficients to be 6\% (27\%); thanks to its strain-derivative nature, the elastocaloric coefficient at \( T_Q \) provides with significantly improved accuracy (1–2\% for both \( \epsilon_{\text{xx}} \) and \( \epsilon_{\text{xx}}^2 \) terms) a view of the evolution of \( T_Q \) with strain.

Following the symmetry-decomposition framework we laid out in Section 4, we assign the \( \epsilon_{\text{xx}} \) and \( \epsilon_{\text{xx}}^2 \) terms in \( T_Q \) to the symmetric (\( \epsilon_{A_{1g}} \)) and antisymmetric (\( \epsilon_{B_{1g}} \)) strain channels, respectively. They are expected to result from leading terms in the free energy of the form \( \epsilon_{A_{1g}} q^2 (\epsilon_{B_{1g}} q^2) \) for \( \epsilon_{A_{1g}} (\epsilon_{B_{1g}}) \), as shown in Fig. 3D (\( q \) represents the staggered quadrupolar order parameter). The absence of an \( \epsilon_{A_{1g}} q^2 \) term in \( T_Q \) is corroborated by the linear pressure-evolution of \( T_Q \) up to hydrostatic pressure of 8 GPa, where the in-plane \( \epsilon_{A_{1g}} \) component \( 1/2(\epsilon_{\text{xx}} + \epsilon_{\text{yy}}) \) reaches \(-0.8\% \) (14, 31). The above symmetry decomposition allows us to conclude that antisymmetric strain suppresses \( T_Q \) in a quadratic manner and that at the highest strain in our experiments (both compressive and tensile), the antisymmetric strain contribution (\( \epsilon_{\text{xx}}^2 \) term) becomes comparable and exceeds the symmetric strain contribution (\( \epsilon_{\text{xx}} \) term).

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\(^1\)The black solid curve is scaled along the temperature axis by a factor of 4, which we hypothesize originates from an imperfect adiabaticity of our experiments.

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**Tetragonal-Symmetry-Breaking in the Canted Antiferromagnetic (CAFM) Phase**

Having identified considerable contributions from both \( \epsilon_{A_{1g}} \) and \( \epsilon_{B_{1g}} \) in the strain-modulation of \( T_Q \), we turn to the lower CAFM phase transition. In Fig. 4A, starting from \( \epsilon_{\text{xx}} \) on the tensile side \( \epsilon_{\text{xx}} = 0.26\% \) (black curve), the peak in \( dT/d\epsilon_{\text{xx}} \) at \( T_N \) sits on top of a continuously varying background, and its height is initially not much varied by decreasing \( \epsilon_{\text{xx}} \); below \( \epsilon_{\text{xx}} = -0.1\% \), however, a downward peak resembling a horizontal mirror image of the peak at \( \epsilon_{\text{xx}} > 0 \) abruptly emerges. This can be contrasted with the continuous variation of the phase transition jumps at \( T_Q \) in Fig. 3A, 3B and C, we trace the location of these peaks in \( dT/d\epsilon_{\text{xx}} \) and the height of the associated jumps \( \Delta(dT/d\epsilon_{\text{xx}}) \), respectively. Due to the presence of multiple singular features at \( \epsilon_{\text{xx}} = -0.2 \sim -0.13\% \), in Fig. 4B and C we mark the peaks with the respective symbol sizes proportional to their peak widths (SI Appendix).

In Fig. 4B, we can identify a “V”-shape in the \( T - \epsilon_{\text{xx}} \) plane with its two branches marked by the primary positive and negative peak features, respectively; this is accompanied by a step-wise structure of \( \Delta(dT/d\epsilon_{\text{xx}}) \) as a function of \( \epsilon_{\text{xx}} \) displayed in Fig. 4C. The “branched” phase boundary structure prompts us to propose the emergence of two distinct sets of domains below \( T_N \) that are strain-selective. Above \( \epsilon_{\text{xx}} = -0.13\% \), a linear fit to \( \Delta(C(dT/d\epsilon_{\text{xx}}))/\Delta C \) (Fig. 4 C, Inset) yields \( A = 66.6(8), B = -3.6 \times 10^2 (A + B \epsilon_{\text{xx}}) \), which via Eq. 4 suggests a linear dependence of \( T_N \) with \( \epsilon_{\text{xx}} \), consistent with our observation (Fig. 4B); we note that the \( B \) coefficient, which via Eq. 4 is associated with a potential \( \epsilon_{\text{xx}}^2 \) term in \( T_N(\epsilon_{\text{xx}}) \), cannot be distinguished from zero and is at least two orders of magnitude smaller than its counterpart in \( T_Q(\epsilon_{\text{xx}}) \).

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\(^2\)To quantitatively compare \( A_{1g} \) with \( T_Q(\epsilon_{\text{xx}}) \) in Fig. 4B, an additional factor 2.3 is required in front of \( A \). We note that this factor introduced to account for imperfect adiabaticity is comparable with that used above for \( T_Q \). The difference between the two factors may arise from a \( T \)-dependence of the thermal conditions (thus the adiabaticity) of the experimental setup.
Below, we discuss the nature of the strain-selective domains and the linear evolution of $T_N$ with $\epsilon$. In the CAFM phase of DyB$_2$C$_2$, it is known that there exists net magnetization $m$ along in-plane [100] directions (19, 35); via magnetoelastic coupling (38), these domains can distort along [100] below $T_N$, providing a natural origin for the proposed strain-selective domains. The lowest order magnetoelastic coupling is required to take the form of $\epsilon m^2$ to be invariant under time-reversal symmetry (38), and we group the four [100] magnetic domains into subgroups with $m$ along $\pm x$ and $\pm y$, as illustrated in Fig. 4D. Additionally taking into account $C_4$ (for discussions hereafter, we still adopt the convention of the high $T$ $D_{4h}$ group), we may decompose $\epsilon m^2$ into terms containing antisymmetric $\epsilon_{B_{1g}}(m_x^2 - m_y^2)$ and symmetric strain $\epsilon_{A_{1g}}(m_x^2 + m_y^2)$, respectively (39). We note that the consequences of the $\epsilon_{B_{1g}}$ term are twofold: It selects among the differently distorted domains, and promotes the order within the favored domains while suppressing that of the other type (both in an $\epsilon$-linear fashion), as we illustrate in Fig. 4E; joining $T_N(\epsilon_{B_{1g}})$ of the favored domains yields a V-shape, akin to that observed in Fig. 4B. We note that similar phase diagrams have been discussed for antisymmetric strain manipulation of in-plane charge density wave orders in tetragonal (Er,Tm)Te$_3$ (10) and in the context of $p_x \pm ip_y$-type multicomponent superconducting phases (3), both with similar forms of order parameter-antisymmetric strain coupling. We note that the deviation of the center of the “V-shape” in Fig. 4B from zero bias strain (nominally defined as the state the system is cooled down to with zero bias voltage on the piezoelectric stacks) may result from a differential thermal contraction between the strain cell and the sample.

In Fig. 5A, we show $dT/d\epsilon_{xx}$ taken in strain scans at selected $T$: above $T_N$ $dT/d\epsilon_{xx}$ appears linear in $\epsilon$ over the measured strain range, while below $T_N$ a nonlinear component develops; below 12 K a strain-hysteresis additionally opens up. Both nonlinearity and hysteresis in $dT/d\epsilon_{xx}(\epsilon_{xx})$ are consistent with $\epsilon$-selectivity of the CAFM domains: We attribute the positive strain limit where the system reaches a constant slope to a state with a single type of orientational domains, which is separated by a multidomain state from a state with the alternative set of orientational domains (see Insets of Fig. 5 A). The domain selection necessarily arises from antisymmetric strain and requires an $\epsilon_{B_{1g}}$ lattice distortion in each of the domains, and is also consistent with strain (or more precisely, one component of the strain tensor), instead of stress, being the thermodynamic quantity that is held constant in our experiments (40).

The symmetry-lowering at the CAFM phase is additionally evidenced by the mapping of thermally modulated optical birefringence shown in Fig. 4 B–D; a similar technique had been used to image nematic domains in other systems (41) (Additional information on the optical setup can be found in SI Appendix). In Fig. 4B, two types of domains with their principal axes near the crystallographic [100] orientations (Fig. 4C and D) can be identified, and their domain wall appears to run approximately 45° between the principal axes. The birefringence imaging confirms the broken $C_4$ with an orthorhombicity developing along [100] below $T_N$, and complements the above thermodynamic evidence of tetragonal symmetry-breaking in the CAFM phase. The observed diagonal domain wall is also consistent with that commonly observed on surfaces of ferromagnets with fourfold easy axes in the plane (42). It has been suggested previously that magnetic order in DyB$_2$C$_2$ is accompanied by slight rotation of quadrupole moments from a staggered arrangement due to a competition between quadrupolar exchange and magnetic exchange interactions, and as a result, the crystalline symmetry is lowered from tetragonal to orthorhombic (35); that
Fig. 5. Tetragonal symmetry-breaking in the CAFM phase of DyB2C2O8. (A) \(\epsilon_{xx}\)-hysteresis of \(dT/\epsilon_{xx}\) at selected \(T\) at zero magnetic field. The schematics at the top illustrate the favored domains on the compressive (left) and tensile (right) strain side, respectively. The pink curve (\(T = 17 K\)) is offset by 30 K for clarity. The gray arrows indicate the direction of the \(\epsilon_{xx}\)-scans in the hysteresis. (B) Map of principal axis orientation over a 130 \(\times\) 130 \(\mu\)m\(^2\) area with a step size of 5 \(\mu\)m with the orientation defined in (C). The dashed line is a guide to the eye outlining an observed domain boundary. (D) Histogram of orientations shown in (B), indicating two domains whose optic axes are offset by 90\(^\circ\) from one another. Measurements in (B-D) were taken at 2.5 K.

the CAFM phase is potentially monoclinic is proposed in a thermal expansion study (43). Our experimental observations at present cannot rule out a monoclinic nature of the CAFM phase, while we can set a bound that the highest point group symmetry of the CAFM phase is orthorhombic with the in-plane principal axes along the high-temperature tetragonal [100] orientations.

Antisymmetric Elastocaloric Effect from Staggered Quadrupole Moments

In the preceding sections, we focused on the strain tuning of \(T_Q\) and \(T_N\); in the following, we examine the overall strain responses in the system not limited to the proximity of the phase transitions. The intertwined magnetic and quadrupolar degrees of freedom motivate us to use a small polarizing magnetic field to eliminate complications from hysteretic and multidomain behaviors. \(dT/\epsilon_{xx}\) obtained with a magnetic field of 0.3 T parallel to [100] (also the stress direction) are summarized in Fig. 6A (see SI Appendix for \(M(H)\) hysteresis of an unstrained sample). We note that the application of \(H\) appears to simplify \(dT/\epsilon_{xx}\) near \(T_N\) as compared to the zero field responses (i.e., the peak does not reverse in sign as a function of \(\epsilon_{xx}\), in contrast to the zero field cases seen in Fig. 4A and SI Appendix, Fig. S6) and that the peak at \(T_N\) in \(H\) is rounded due to the presence of a finite net magnetization. \(dT/\epsilon_{xx}\) at different \(\epsilon_{xx}\) in Fig. 6A appear to be composed of an \(\epsilon_{xx}\)-independent contribution to \(dT/\epsilon_{xx}\) (which can be approximated by the blue trace at \(\epsilon_{xx} = 0.01\%)\) and a component that varies monotonically with \(\epsilon_{xx}\). In Fig. 6B, we examine constant-\(T\) \(\epsilon_{xx}\) cuts of \(dT/\epsilon_{xx}\). The \(\epsilon_{xx}\)-linear dependence of \(dT/\epsilon_{xx}\) over the entire strain-range at all \(T\) in Fig. 6B can be contrasted with the nonlinear \(dT/\epsilon_{xx}\) in Fig. 5A, and is consistent with a single domain state below \(T_N\) in field. Results of linear fits to \(\epsilon_{xx}\)-cuts of \(dT/\epsilon_{xx}\) at 0.3 T are summarized in Fig. 6C; in Fig. 6C, we also include both the intercept and slope extracted from zero field \(dT/\epsilon_{xx}\) above \(T_N\) as dark blue circles: The close comparison between 0 T and 0.3 T responses above \(T_N\) is consistent with the time-reversal-symmetric nature of the quadrupolar order. That the \(T\)-trace of the intercept of \(dT/\epsilon_{xx}\) (Fig. 6 C, Inset) compares closely to the responses at \(T_N\) on the tensile strain side at zero field (Fig. 4A) suggests that the magnetic domain favored by \(H\) coincides with that favored by tensile \(\epsilon_{xx}\), from which we infer that the long axis of the distorted unit cell is along \(m\) (Fig. 6 A, Inset). Hereafter we focus on the \(\epsilon_{xx}\)-slope of \(dT/\epsilon_{xx}\) (main panel of Fig. 6C). As we invoke above, the \(\epsilon\)-odd component in \(dT/\epsilon\) by symmetry originates from antisymmetric strain (Fig. 1G), which in the present case is \(\epsilon_{B_{zz}}\); Viewed alongside Eq. 1, above \(T_Q\), \(d^2T/\epsilon d\epsilon^2 > 0\) indicates a quadratic decrease of \(S\) with \(\epsilon_{B_{zz}}\), consistent with strain suppression of quadrupolar fluctuations; similar \(\epsilon\)-dependence of \(S\) has been reported in iron-based superconductors above the nematic phase transition and attributed to a bilinear coupling between antisymmetric strain and underlying nematic fluctuations (28). Below \(T_Q\), \(d^2T/\epsilon d\epsilon^2 < 0\) implies on the contrary a quadratic increase of \(S\) with \(\epsilon_{B_{zz}}\). In Fig. 6D, we show a contour plot of the entropy landscape with \(\epsilon_{B_{zz}}\) inferred from the slope in Fig. 6C and the zero strain heat capacity (see SI Appendix for the procedure to extract \(S(\epsilon, T)\) and thus deduce \(C(\epsilon, T)\) (27)) near \(T_Q\), where a curvature change in the entropy landscape across \(T_Q\) is apparent.

To shed light on the antisymmetric strain effects on the AQF order and the underlying staggered quadrupolar arrangement, we
introduce the following Hamiltonian

\[ H = K \sum_{<ij>} Q_i Q_j - g \epsilon_{Bg} \sum_i O_i \]  

[5]

where the first term describes the quadrupole-quadrupole interaction between the nearest neighbors \((K > 0 \text{ gives an AFQ order})\) and the second term the quadrupole-strain coupling with \(g > 0\) \((13, 44)\). Eq. 5 can be further mapped onto an effective AFM Ising model with the quadrupole moments \(O\) mapping onto Ising spins \(S^z\) and \(\epsilon_{Bg}\) mapping onto an effective magnetic field \(H_z (J > 0)\) (Fig. 7A):

\[ H = J \sum_{<ij>} S^z_i S^z_j - H_z \sum_i S^z_i , \]  

[6]

whose mean-field entropy landscape is shown in Fig. 7B (here we also include a field-independent phonon background, see SI Appendix). In Fig. 7B, \(H_z\) appears to suppress the critical temperature of the AFM order (similar to \(\epsilon_{Bg}\) for the AFQ order), near which the curvature of the isentropic contours exhibits a sharp change, giving rise to a sign reversal of the antisymmetric slope (SI Appendix) similar to that observed experimentally in Fig. 6 C and D. The comparison suggests that within the ordered state, the antisymmetric strain \(\epsilon_{Bg}\) in this case destabilizes and therefore suppresses the staggered AFQ order akin to how magnetic field destabilizes a staggered Ising antiferromagnetic order by exciting pseudospin flips (quadrupole flops). The nonmonotonic shape of the phase boundary in the \((T, H_z)\) plane near \(T = 0\) has been discussed before for AFM Ising models and attributed to an order-by-disorder effect (45, 46). We note that the effective AFM Ising model has only taken into account the quadrupolar degrees of freedom; the continuity of the observed antisymmetric response above and below \(T_N\) aside from a small kink at \(T_N\) in Fig. 6C implies that the overall staggered quadrupole configuration is likely not fundamentally modified (a weak relative reorientation of the quadrupole moments has been
suggested by ref. 35) by the magnetic order and is the source of the observed $\epsilon$-linear responses in $dT/d\epsilon$.

Discussion and Summary

In summary, we have employed the AC elastocaloric effect to investigate the strain responses of the $f$-electron antiferroquadrupolar order in DyB$_2$C$_2$. The strain-dependence of both the quadrupolar and magnetic phase transitions in the system can be precisely characterized by the jump of the elastocaloric signals—the former contains both linear and quadratic strain-dependences while the latter remain linear over the explored strain range. While symmetric strain $\epsilon_{B_{1u}}$ always appears to tune both $T_N$ and $T_Q$ in a linear manner, the antisymmetric strain $\epsilon_{B_{1g}}$, which is inaccessible in hydrostatic pressure experiments, plays an indispensable and more versatile role in controlling $T_Q$ and $T_N$: for the AFQ phase, $\epsilon_{B_{1g}}$ suppresses $T_Q$ in a quadratic manner, while for the CAFM order, the primary role of $\epsilon_{B_{1g}}$ is found to be twofold: domain selection as well as linearly tuning $T_N$. The distinct behavior of the two phase transitions with $\epsilon_{B_{1g}}$ lies in the different forms of coupling between $\epsilon_{B_{1g}}$ and the underlying order parameters $\epsilon_{B_{1g}} g^2$ and $\epsilon_{B_{1g}} (m_x^2 - m_y^2)$ (Figs. 3D and 4F); in this context, we may in turn use the evolution of critical temperatures with antisymmetric strain to place strong constraints on the spatial/lattice symmetry of the underlying order parameters given a generic phase transition.

From a symmetry perspective, $f$-electron-based quadrupolar orders can be viewed as a close analogue of the nematic phases observed in a number of transition-element-based strongly correlated electron systems (18, 47). The relatively well-localized nature of the electronic degrees of freedom and the strong magnetoelastic coupling of $f$-electrons marks them as model systems to drive quantum phase transitions with strain. For instance, it has been proposed that antisymmetric strain orthogonal to a globally uniform ferroquadrupolar order couples to the latter as an effective transverse field, therefore promoting quantum fluctuations and ultimately driving a quantum phase transition into the Ising nematic order (20). In the present case of an antiferroquadrupolar order, we demonstrate that the antisymmetric strain suppresses the AFQ phase transition, likely through introducing quadrupole flops (pseudospin flips) as an effective “longitudinal field” in the AFM Ising model (we note that we do not exclude transverse-field-like effects akin to those proposed in ref. 20). Extrapolating from the measured $T_Q(\epsilon_{B_{1g}})$, we expect that antisymmetric strain on the order of 3–4% may be required to completely suppress the AFQ order and drive a quantum phase transition in the present system. Our study provides a proof-of-principle example of employing antisymmetric strain as a means of driving quantum phase transitions in spatially varying anisotropic electronic orders beyond a uniform rotation–symmetry–breaking nematic order; as briefly discussed in the introduction, examples of systems to which we can extend the above study include orbital order (21, 22), spin and charge stripe order (23, 24), and “hidden,” multipolar orders (25).

Viewed alternatively from the perspective of employing elastocaloric effect as a tool to study strain responses, our results establish that the elastocaloric coefficients provide a refined picture of strain-evolution of given phase transitions, via a modified Ehrenfest relation thanks to its thermodynamic and strain-derivative nature. Additionally, our case study here demonstrates that extending the elastocaloric measurement over a range of strain values provides a pathway to systematically extract effects from spatially symmetric and antisymmetric strains; we anticipate that this framework can be applied as a powerful organizing principle for exploring strain responses of extended classes of quantum materials.

Materials and Methods

Single-Crystal Growth and Characterization. The experiments reported in this paper were performed using high-quality single-crystal samples of DyB$_2$C$_2$. Polycrystalline DyB$_2$C$_2$ was first prepared by melting together Dy ingots, B pellets and graphite rods in a molar furnace. Single crystals of DyB$_2$C$_2$ were then grown using the Czochralski method in a tetra arc furnace (GES Corporation, TAC-35325) in an Ar atmosphere. Typical current used during the growth is 25 A per arc, and single crystalline rods were pulled out from the melt at a rate of approximately 4 mm/h. The obtained rods cleave along (001) planes, and we confirmed the phase and orientation of the obtained crystals using X-ray diffraction. Magnetization measurements were performed in a Quantum Design MPMS-3 SQUID Magnetometer.

Elastocaloric Measurements. Elastocaloric measurements at cryogenic temperatures were performed using a Razorbill CS-100 uni axial stress cell in a Quantum Design Physical Property Measurement System (PPMS). The crystals were shaped into long rectangular plates and attached to Ti plates using Stycast FT-2850 cured at 80°C for at least 3 h. In main text Fig. 2B, we show an optical image of a DyB$_2$C$_2$ crystal used in the elastocaloric measurements. The single crystalline piece of DyB$_2$C$_2$ is mounted on the sample cell with the uniaxial stress applied along the in-plane [100] direction. The sample dimension was 2.66 mm × 0.43 mm × 0.032 mm, and the effective strained length between the two Ti mounting plate is 1.628 mm. The temperature oscillations were measured using either an Au(0.07%Fe)-chromel thermocouple or a RuO$_2$ thermometer (for clarity, the experimental data shown in the main text are all obtained from the latter); the former is attached to the sample via a thin layer of AngstromBond ABY110LV (Fiber Optic Center) while the latter is thermally connected to the sample via a 50 µm gold wire. Additional silver paste (DuPont 4929N) is applied onto the gold wire to enhance the thermal coupling between the sample and the thermometer. The thermometer near the sample is connected to three identical thermometers thermally anchored at the bath forming a full Wheatstone bridge to single out the temperature oscillation in response to the applied AC strain. In the experiments, an AC strain with a frequency $\nu$, is applied through the outer stack of CS-100, and the sine-out of phase of lock-in amplifier SR860 (Stanford Research Systems) is first amplified with TEGAM-2350 High-Voltage Power Amplifier (by a factor of 50) before reaching the piezoelectric stacks. The inner piezoelectric stack is powered through an auxiliary DC output of SR860 amplified via an SVP 150 bipolar/1 High-Voltage Amplifier (Piezomechanik) by a factor of 50. Typical AC strain oscillation amplitude used in the experiments is approximately $5 \times 10^{-5}$. The temperature (voltage) responses from the thermocouple are captured at the frequency $\nu$, while that from the thermometer is captured at the frequency ($\nu$ - $\nu$) where $\nu$ is the AC current excitation applied to the bridge circuit. We used current frequency of 1.4 kHz and AC strain frequency of 27.1 Hz in data shown in Figs. 2-5 and 6 A and B while the low temperature ($T < 12$ K) slope in Fig. 6C are extracted from measurements taken with AC strain frequencies adjusted for each temperature to account for the shifting optimal frequency windows (SI Appendix).

Mechanical State in the Elastocaloric Experiments. In our experiments, the longitudinal strain $\epsilon_{xx}$ is the thermodynamic control parameter, and the sample is under such a mechanical state that the only finite component in the stress tensor $\sigma_{ij}$ is $\sigma_{xx}$. The measured elastocaloric coefficient is denoted as $(dT/d\epsilon_{xx})$ for simplicity while we note that it is a linear combination of the following partial derivatives multiplied by the respective Poisson ratios:

$$\frac{dT}{d\epsilon_{xx}} = \left(\frac{\partial T}{\partial \epsilon_{xx}}\right)_{\epsilon_{yy}} + \left(\frac{\partial T}{\partial \epsilon_{yy}}\right)_{\epsilon_{xx}} + \left(\frac{\partial T}{\partial \epsilon_{zz}}\right)_{\epsilon_{xx}} + \left(\frac{\partial T}{\partial \epsilon_{xy}}\right)_{\epsilon_{xx}}.$$  [7]
The Poisson ratios between $\epsilon_{xx}, \epsilon_{yy}, \epsilon_{zz}$ under our experimental condition can be solved from the following set of linear equations through the elastic moduli tensor of LuB$_2$Cu$_2$ (32):

$$
\begin{bmatrix}
\sigma_{xx} \\
0 \\
0
\end{bmatrix} =
\begin{bmatrix}
525 & 243 & 34 \\
243 & 525 & 34 \\
34 & 34 & 224
\end{bmatrix}
\begin{bmatrix}
\epsilon_{xx} \\
\epsilon_{yy} \\
\epsilon_{zz}
\end{bmatrix},
$$

from $\sigma_{yy} = \sigma_{zz} = 0$ we obtain $\nu_{13} = -\epsilon_{xx}/\epsilon_{yy} = 0.08$, $\nu_{12} = -\epsilon_{yy}/\epsilon_{xx} = 0.45$. Eq. 7 can be alternatively expressed in its symmetric and antisymmetric components (here $\epsilon_{xx} + \epsilon_{yy}$ and $\epsilon_{xx} - \epsilon_{yy}$):

$$
\begin{align*}
\frac{dT}{d\epsilon_{xx}} &= \left( \frac{\partial T}{\partial \epsilon_{xx}^2 + \epsilon_{yy}^2} \right) \frac{d\epsilon_{xx}^2 + \epsilon_{yy}^2}{d\epsilon_{xx}} + \left( \frac{\partial T}{\partial \epsilon_{xx} \epsilon_{yy}} \right) \frac{d\epsilon_{xx} \epsilon_{yy}}{d\epsilon_{xx}}, \\
&+ \left( \frac{\partial T}{\partial \epsilon_{xx}^2 - \epsilon_{yy}^2} \right) \frac{d\epsilon_{xx}^2 - \epsilon_{yy}^2}{d\epsilon_{xx}}
\end{align*}
$$

along $T(\epsilon)$. This suggests that

$$
\frac{dT_C}{d\epsilon} = \left. \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{xx}} - \left. \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{yy}}.
$$

Using $TdS = C_T dT$ and Eq. 1 we get

$$
\frac{dT_C}{d\epsilon} = C_1 \left( \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{xx}} - \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{yy}} C_2,
$$

where $C_1$ and $C_2$ are the heat capacity of the two phases at $T_C$, Eq. 12 may be reformulated as

$$
\frac{dT_C}{d\epsilon} = \left. \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{xx}} - \left. \frac{\partial T_C}{\partial \epsilon} \right|_{\epsilon = \epsilon_{yy}}
$$

analogous to the Ehrenfest relation relating the phase boundary with respect to uniaxial pressure $T_C(P)$ to the jumps at thermal expansion $\alpha_i$ and heat capacity (37):

$$
\frac{dT_C}{d\epsilon} = \frac{\Delta C(1/T_C(\epsilon))}{\Delta T}.
$$

Here $\Delta T$ is the molar volume.

The above derivation is based on a generic form of applied $\epsilon$. Taking into account the experimentally relevant linear combination of strain modes discussed in the main text, the modified Ehrenfest relation in the context of our experiments can be expressed as

$$
\frac{dT_C}{d\epsilon} \right|_{\epsilon = \epsilon_{xx}} = \frac{\Delta C(1/T_C(\epsilon))}{\Delta T}.
$$

Data, Materials, and Software Availability. Spreadsheet data have been deposited in Stanford Digital Repository (https://doi.org/10.25740/wy53mp2837) (36).

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