Sr$_2$RuO$_4$ (SRO$_{214}$) is a prototypical unconventional superconductor. However, since the discovery of its superconductivity a quarter of a century ago, the symmetry of the bulk and surface superconducting states in single crystal SRO$_{214}$ remains controversial. Solving this problem is massively impeded by the fact that superconducting SRO$_{214}$ is extremely challenging to achieve in thin-films as structural defects and impurities sensitively annihilate superconductivity. Here we report a protocol for the reliable growth of superconducting SRO$_{214}$ thin-films by pulsed laser deposition and identify universal materials properties that are destructive to the superconducting state. We demonstrate that careful control of the starting material is essential in order to achieve superconductivity and use a single crystal target of Sr$_3$Ru$_2$O$_7$ (SRO$_{327}$). By systematically varying the SRO$_{214}$ film thickness, we identify mosaic twist as the key in-plane defect that suppresses superconductivity. The results are central to the development of unconventional superconductivity.
The past decade has seen rapid developments in the understanding of unconventional superconductivity, particularly in proximity-coupled systems involving conventional s-wave superconductors in combination with magnetic materials and interfaces with strong spin-orbit coupling. Highlights include the discovery of odd-frequency (s-wave) spin-triplet pairing at s-wave superconductor/ferromagnetic interfaces\(^2\),\(^3\), evidence for electron-composite particle-antiparticles in nanowire devices with spin-orbit coupling and superconductivity\(^1\),\(^11\),\(^12\), and surface superconductivity in Au with Fermi-level tuning via a ferromagnetic dielectric\(^13\).

Parallel research on intrinsic unconventional superconductivity in superfluid He and in compounds such as Sr\(_2\)RuO\(_4\) (SRO\(_{214}\))\(^14\),\(^15\) has also made dramatic advances. Single crystal SRO\(_{214}\) has a superconducting critical temperature\(^16\) (\(T_c\)) of 1.5 K. Although the underlying nature of the superconducting state in SRO\(_{214}\) crystals remains highly controversial, the consensus from experiments and theory is that the pairing is unconventional and potentially chiral p-wave state with the d-vector perpendicular to the basal plane, which is even-frequency and conceptually different from the odd-frequency spin-triplet pairing induced at s-wave superconductor/ferromagnetic interfaces. Muon spin-relaxation measurements\(^3\),\(^19\) show evidence for time-reversal symmetry breaking below \(T_c\) while early nuclear magnetic resonance spectroscopy\(^20\),\(^21\) and polarized neutron scattering\(^22\) experiments have demonstrated a constant in-plane spin susceptibility (Knight shift) below \(T_c\). However, a constant out-of-plane spin susceptibility below \(T_c\) goes against a chiral p-wave state\(^2\). Furthermore, recent nuclear magnetic resonance spectroscopy on SRO\(_{214}\) crystals show that the Knight shift decays in the superconducting state in the “3 K phase” under uniaxial stress as well as in the “1.5 K phase” without stress\(^23\), consistent with a d-wave or helical p-wave state.

Although there is a lack of experimental consistency in the underlying superconducting symmetry of SRO\(_{214}\), unconventional pairing states are expected on the surface due to broken inversion symmetry, which raises the prospect of coupling different superconducting symmetries via proximity effects with a fixed number of laser pulses (5000) at 2 Hz and laser fluence of 1.0 J cm\(^{-2}\) to achieve a thickness (\(d\)) around 23 nm (depending on \(t\)). Values of \(t\) are estimated by fitting thickness fringes to the (0 0 6) diffraction peak of SRO\(_{214}\) (see Supplementary Note 1 and Supplementary Fig. 3). In Fig. 1c we have plotted X-ray diffraction fringes from three representative SRO\(_{214}\) films grown using 1.0 Pa, 0.35 Pa and 0.09 Pa of oxygen. For the \(P_{O_2}=1.0\) Pa film, thickness fringes are barely visible on the (0 0 6) peak and an SRO\(_{214}\) impurity phase is present (highlighted in green). The \(P_{O_2}=0.35\) Pa film, however, shows no detectable evidence for SRO\(_{214}\) or other impurity phases and the (0 0 6) peak shows clear fringes, indicating uniform growth. Reducing \(P_{O_2}\) further to 0.09 Pa promotes secondary phases in the Ruddlesden-Popper series (indicated by magenta) such as SRO\(_{327}\), SRO\(_{413}\), or RuO\(_2\). While thin films deposited above or below \(P_{O_2}=0.35\) Pa show metallic behavior with no detectable evidence for superconductivity down to 300 mK, the \(P_{O_2}=0.35\) Pa film shows a downturn in \(R(T)\) below 0.5 K, consistent with the onset of incipient superconductivity (Supplementary Fig. 4a). The corresponding lattice parameters are \(a = 0.3870(3)\) nm (from RSM on (2014)\(^24\) of SRO\(_{214}\) have been grown by molecular beam epitaxy on LSAT with a \(T_c\) of 1.1 K using a Ru-rich flux during growth to reduce Ru loss\(^30\). The \(T_c\) was further enhanced to 1.9 K by depositing onto single terminated (1 1 0) NdGaO\(_3\) due to the associated misfit strain\(^31\).

In this article we set out to develop a protocol for the reliable growth of superconducting SRO\(_{214}\) thin-films by pulsed laser deposition and to establish and control key materials properties that are destructive for superconductivity. This is achieved using careful control of the starting material, which consists of a single crystal target of Sr\(_2\)RuO\(_4\) (SRO\(_{327}\)). We establish that mosaic twist is a universal structural (in-plane) defect that destroys superconductivity in SRO\(_{214}\). This is clearly different from the planar defects (out-of-plane boundaries) reported by Krockenberger\(^32\). By controlling the degree of mosaic twist in the SRO\(_{214}\) films, and the associated dislocations that form at the SRO\(_{214}\)/substrate interface, we demonstrate a reliable protocol for depositing superconducting SRO\(_{214}\).

### Results

**Growth optimization of SRO\(_{214}\) thin films.** A single crystal target of SRO\(_{227}\) provides 33% Ru excess that compensates for Ru loss during the high-temperature growth of SRO\(_{214}\) which, in conjunction with fine-control of laser fluence\(^32\) (see Supplementary Fig. 1 for results using a polycrystalline target of SRO\(_{214}\)), reduces the volume fraction of impurity phases present in the films. The single crystal target of SRO\(_{227}\) (see compositional analysis in Supplementary Fig. 2a) has a surface area of \(3 \times 10^{-10}\) mm\(^2\) and is mounted on a polycrystalline SRO\(_{214}\) holder (Fig. 1a). Laser alignment is optimized by focusing the laser spot (1.5 mm diameter) onto the SRO\(_{214}\) holder prior to ablating from the SRO\(_{227}\) target. During film growth, the rotation of the SRO\(_{227}\) target is fixed and the target carousel is twisted through an angle \(\beta\) to erode the SRO\(_{227}\) target along a line (Fig. 1b). All SRO\(_{214}\) films discussed in this paper are deposited onto heated (950°C) LSAT (0 0 1) as described in the Methods section. Most of the films are grown using a laser pulse frequency of 2 Hz with one sample grown at 4 Hz as discussed at the end of the paper. To minimise in-plane epitaxial strain, SRO\(_{214}\) (in-plane lattice constant = 0.3873 nm) is grown on LSAT (in-plane lattice constant = 0.387 nm) substrates, and a substrate miscut angle of less than 0.05° is used to reduce the concentration of out-of-plane stacking faults at step edges.

We first discuss the effect of varying oxygen pressure (\(P_{O_2}\)) during growth. SRO\(_{214}\) films are grown using a fixed number of laser pulses (5000) at 2 Hz and laser fluence of 1.0 J cm\(^{-2}\) to achieve a thickness (\(d\)) around 23 nm (depending on \(P_{O_2}\)). Values of \(t\) are estimated by fitting thickness fringes to the (0 0 6) diffraction peak of SRO\(_{214}\) (see Supplementary Note 1 and Supplementary Fig. 3).
214, 327 and 4310 refer to Sr2RuO4, Sr3Ru2O7 and Sr4Ru3O10, respectively.

Different laser mounted on a polycrystalline Sr2RuO4 holder.

The peak marked “β” is labelled the pulsed laser deposition setup, where the rotation of the target carousel corresponds to the diffraction plane of the Ruddlesden-Popper series (0 0 l) (see Methods). We compare STEM (Fig. 3a, g) and HR-STEM micrographs which demonstrate coherent c-axis growth (Fig. 3b, l). For those films that show a full superconducting transition, the micrographs reveal inclusions near the SRO214/LSAT interface (Fig. 3a). These crystalline (Fig. 3f) inclusions are elemental Ru (Fig. 3c–e) or Ru oxide (Supplementary Fig. 7) and spaced over distances larger than ξ, and so should not directly affect Tc.

For the SRO214 films that do not show a full superconducting transition, STEM maps consistently reveal a 1–2-nm-thick region above LSAT (indicated with an arrow) that has mixed stoichiometry (Fig. 3g). STEM-EDX confirms that this region has the correct Sr atomic concentration for SRO214 but is deficient in Ru and rich in O (Fig. 3i–k). This is further confirmed by HR-STEM on different areas of the film (Fig. 3h,l), which show atomic layers of decreased concentration of heavy atoms, and stoichiometric SRO214 layers above and below this region (Fig. 3g).

Characterization of superconducting properties. We now discuss electrical transport of SRO214 films versus t in the 15 nm to 166 nm range using optimized growth parameters (oxygen pressure of 0.35 Pa and fluence of 1.0 J cm−2). For each film we investigate R(T) and for those that show a superconducting transition, we define Tc as the temperature halfway through the resistive transition. In Fig. 2a we have plotted Tc(t) where the vertical error bars represent the temperature width of the superconducting transition (Supplementary Fig. 5). These data show a critical thickness for superconductivity of approximately 50 nm with Tc rising to 1.05 K for t = 166 nm (Fig. 2a).

Although the largest Tc is lower than the Tc of bulk SRO214 single crystals (1.5 K), Tc values are higher than previous reports for SRO214 films grown by pulsed laser deposition (Supplementary Fig. 6a).

In Fig. 2b we have plotted the t-dependence of the residual resistivity ratio (RRR), defined at the resistance at room temperature divided by the saturated minimum in resistance at low temperature before the onset of superconductivity. RRR(t) broadly divides into two regimes: for t < 50 nm, RRR is low (<30) with metallic transport down to 300 mK (highlighted in red); for t > 50 nm, RRR rapidly increases with increasing t with superconducting transport at low temperature (highlighted in blue). The low RRR(t) values in the metallic regime cannot be simply explained on the basis of a thin-film effect or t approaching the out-of-plane superconducting coherence length of SRO214, which we estimate to be ξc ~3–8 nm (see Fig. 2c and Supplementary Note 2). The metallic regime indicates a large density of defects due to impurity phases in conjunction with structural defects (e.g. mosaic tilt or mosaic twist), consistent with the high (low) values of residual resistivity (ρ0) for the films with a low (high) RRR as shown in Fig. 2d.

In the superconducting regime, RRR reaches 110 for t = 100 nm, which is high relative to equivalently-thick SRO214 films reported elsewhere (Supplementary Fig. 6b).

Analysis of the microstructure. To identify the underlying mechanisms which suppress superconducting behavior in SRO214 films, we have systematically investigated the potential presence of structural defects that may affect long-range crystal order. We first discuss scanning transmission electron microscopy (STEM), high resolution scanning transmission electron microscopy (HR-STEM) and energy-dispersive X-ray (EDX) maps acquired on a SRO214 superconducting film (Fig. 3a–f) and a metallic film (Fig. 3g–l) (see Methods). We compare STEM (Fig. 3a, g) and HR-STEM micrographs which demonstrate coherent c-axis growth (Fig. 3b, l). For those films that show a full superconducting transition, the micrographs reveal inclusions near the SRO214/LSAT interface (Fig. 3a). These crystalline (Fig. 3f) inclusions are elemental Ru (Fig. 3c–e) or Ru oxide (Supplementary Fig. 7) and spaced over distances larger than ξ, and so should not directly affect Tc.

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Fig. 1 Pulsed laser deposition setup and structural properties of Sr2RuO4 thin films. a A photograph showing a single crystal Sr2Ru2O7 target mounted on a polycrystalline Sr2RuO4 holder. b A schematic illustration of the pulsed laser deposition setup, where the rotation of the target carousel is labelled “β”. c, d X-ray diffraction traces for Sr2RuO4 grown at 950°C under different O2 pressures (labelled) with a fixed fluence of 1.0 J cm−2 (c) and equivalent traces in which Sr2RuO4 is grown at 950°C using different laser fluxes (labelled) with a fixed oxygen pressure of 0.35 Pa (d). The diffraction planes for Sr2RuO4 (orange) SRO327 (green) and (La0.3Sr0.7)(Al0.65Ta0.35)O3 (LSAT) (blue). The peak marked “ω” corresponds to the diffraction plane of the Ruddlesden-Popper series (0 0 l). 0 0 1327, 0 0 14 4310 or 2 1 0) diffraction plane of RuO2. The subindexes 214, 327 and 4310 refer to Sr2RuO4, Sr3Ru2O7 and Sr4Ru3O10, respectively.

plane) and c = 1.2738(17) nm (from a 2θ–ω scan on the (0 0 l) peak positions after applying a correction for sample displacement).

Using P02 = 0.35 Pa, we now discuss the effect of laser fluence on the structural and electrical properties of SRO214. In Fig. 1d we have plotted X-ray diffraction traces from three films grown using laser fluences of 0.75 J cm−2, 1.0 J m−2 and 1.4 J cm−2, with a fixed number of laser pulses (5000). The traces show sharper peaks with decreasing laser fluence, indicating improved structural properties (vertical microstrain). The in-plane resistance versus temperature R(T) for the 1.4 J cm−2 film saturates to a constant minimum below 10 K with no evidence for superconductivity down to 300 mK. In contrast to the 1 J cm−2 film, the 0.75 J cm−2 film shows sharper diffraction peaks, but a downturn in R(T) is not observed, even down to 300 mK (Supplementary Fig. 4b). This is likely due to the lower laser fluence reducing Ru ablation from the SRO327 target, and hence, the SRO214 film is deficient in Ru, which prevents superconducting behaviour29,30.
The possible SRO$_{327}$ phase would be concentrated at the SRO$_{214}$/LSAT interface in the initial growth, considering the thickness dependence of our superconducting films. Such intergrowth could be controlled later by changing the dynamic nature of PLD. Further studies are highly desirable. Additionally, since STEM-EDX confirms stoichiometric Ru content on the SRO$_{214}$ thin films (see example in Supplementary Fig. 2b), our study focuses on the analysis of structural defects that could alter the superconducting transition.

**Degree of tilt and twist.** Another potential source of crystallographic defects that may suppress superconductivity relate to misoriented crystalline regions. This can be visualized with the mosaic crystal model, in which the film is described as the combination of smaller crystallites (blocks), misoriented with respect to each other and the substrate. A rotation of these blocks about an axis parallel to the surface is known as mosaic tilt, and a rotation about an axis perpendicular to the surface is known as mosaic twist. Tilted and twisted blocks are separated by low-angle dislocation boundaries consisting of dislocations, which can be edge- or screw-like, with a Burgers vector perpendicular or parallel to the dislocation line vector (u), respectively (see Supplementary Fig. 9), and cause local variations of interplanar distance (microstrain) at grain boundaries. Tilt can be measured from the full width half maximum in omega (FWHM$_{\omega}$) of the (0 0 1) diffracting planes by X-ray diffraction in a symmetric (coplanar) geometry. Values of tilt extracted from SRO$_{214}$ films with different $t$ show that all films (superconducting or non-superconducting), with the exception of one, have similar values of tilt. Tilt does not therefore affect the electrical properties SRO$_{214}$ films (see Supplementary Fig. 10a–c).
Twist can be measured using four different configurations: edge [by measuring the \((h\ k\ 0)\) planes from the FWHM\(_n\) with an offset in chi (\(\chi_{\text{offset}}\)) of 90° (Fig. 4a)]; glancing angle in-plane; transmission; or skew geometry\(^{37}\). For our thin-film geometry we adopt the skew geometry (non-coplanar) configuration because the signal intensity is the strongest, and measure the FWHM\(_\omega\) of the (4 1 3) diffracting planes, which provides a good estimate of the degree of twist due to the large \(\chi_{\text{offset}}\) \(>70°\)^{37,38}. The dependence of twist, FWHM\(_n\) extracted from a pseudo-Voigt profile fit after the subtraction of the instrumental contribution (see Supplementary Note 3), on \(t\) is shown on Fig. 4a. Since the in-plane crystallite size \(L_\parallel\) is of the order of micrometres, its contribution to peak broadening is negligible (see Supplementary Fig. 10a). We observe that FWHM\(_n\) (Fig. 4b inset), and hence the degree of twist, rapidly rises as \(t\) decreases below 50 nm, corresponding to the metallic films that do not show full superconducting transition (Fig. 4b). From Fig. 4c we demonstrate a direct correlation between twist and the suppression of superconductivity by a reduction (increase) of \(T_c\) (residual resistivity) when the degree of twist increases.

Identification of dislocations. The increase in twist with decreasing \(t\) (Fig. 4b) indicates a higher concentration of dislocations with a plane in-plane at low \(t\) for non-superconducting films. To confirm this and determine the nature of the dislocations, we have performed \(g\ \cdot\ b\) TEM analysis, with \(g\) being the diffracted beam direction on a superconducting and non-superconducting film. In \(g\ \cdot\ b\) TEM analysis, dislocations are in-contrast (visible) when \(g\ \cdot\ b\neq 0\), but out-of-contrast (invisible) for \(g\ \cdot\ b=0\) as illustrated in Fig. 4a. Figure 4d–o shows \(g\ \cdot\ b\) bright field TEM analysis performed on three different cross-sectional areas for the superconducting and non-superconducting films using two perpendicular diffraction vectors \(g_{001}\) and \(g_{000}\). The non-superconducting films shows a larger concentration of dislocations compared to the superconducting film. Furthermore, for the non-superconducting film the dislocations are mostly in-plane and screw-like with both a and \(b\) in-plane (horizontal orange arrow), as they can only be resolved when \(b\parallel g_{001}\) (Fig. 4d–f) and are extinct when \(b\perp g_{001}\) (Fig. 4g–i). These results are consistent with the high degree of twist observed in non-superconducting films and therefore demonstrate that horizontal screw dislocations are a key defect that strongly suppresses superconductivity in SRO\(_{214}\). In contrast, the lower density of dislocations present in the superconducting film are in-plane edge-like with both \(u\) in-plane and \(b\) out-of-plane (vertical orange arrow), as they are visible when \(b\parallel g_{001}\) (Fig. 4j–l) and not visible when \(b\perp g_{001}\) (Fig. 4m–o). In both the superconducting and non-superconducting films, a few threading mixed dislocations, with both edge and screw components (tilted orange arrow) are resolved with both \(g_{001}\) and \(g_{000}\). The presence of threading dislocations with a screw component is also revealed in topographic images acquired using an atomic force microscope (Supplementary Fig. 10d–g).
We note that, we cannot confirm that the white contrast in Fig. 3j located in the Ru-deficient layer (confirmed in Fig. 4g–i), is also dislocation related as it might be the result of a combination of features such as Ru-deficiency, interface effect and dislocations.

Finally, we note that the superconductivity in SRO214 can be further optimized by tuning additional growth parameters (not discussed in this paper) such as deposition frequency. We have tested the effect of doubling the laser pulse frequency to 4 Hz during SRO214 growth, while keeping the same growth conditions as for the rest of the study (oxygen pressure of 0.35 Pa, fluence of 1.0 J cm$^{-2}$, 950 °C), which has the effect of reducing the degree of twist (Fig. 4b) and the residual resistivity (Fig. 2d), and increasing $T_c$ (Fig. 3a) and RRR (Fig. 3b), compared to equivalent films grown at 2 Hz.
Fig. 4 Nature of dislocations and their effect on superconductivity in Sr2RuO4 films. a An illustration of an edge dislocation with an out-of-plane line vector \( \mathbf{u} \) (grey arrow) and Burgers vector \( \mathbf{b} \) in-plane (orange arrow) on the \((h\,0\,0)\) planes of SRO214. The diagram also shows the X-ray diffraction setup in edge geometry (Tilt angle of the sample stage \( \chi_{\text{offset}} = 90° \)) to measure the in-plane misorientation, twist, from the peak broadening in \( \omega \) (angle between the X-ray incident beam and the diffracted planes) due to variations in the scattering direction (white and yellow arrows). Transmission electron microscopy TEM g•b analysis to reveal edge dislocations using bright field imaging with two perpendicular diffraction conditions \( \{g_{\text{conf}} \text{ and } g_{\text{conf}} \} \) is also shown. b Degree of twist versus thin-film thickness \( t \), determined from the full width half maximum in \( \omega \) FWHM, measured from \( \chi_{\text{offset}} = 78° \) in skew symmetric on the \( (4\,1\,3) \) planes (inset curves are vertically offset for clarity). c, Superconducting critical temperature \( T_c \) versus and resistivity \( \rho \) versus twist (inset). d–g. b Analysis by bright field TEM showing dislocations on three areas of a lamella from metallic (d–f) and superconducting (j–o) SRO214 films (marked as three asterisks in b and c). The diffraction vectors \( \mathbf{g}_{\text{conf}} \) and \( \mathbf{g}_{\text{conf}} \) are indicated by green arrows and \( \mathbf{b} \) by orange arrows: for in-plane edge dislocations the arrows are horizontal, for out-of-plane screw dislocations the arrows are vertical, and for mixed dislocation the arrows are tilted. The interface film/substrate interface is marked with a yellow dotted line. Scale bar in d–o, 50 nm.

Discussion
In conclusion, we have systematically investigated the structure-electrical-properties relationship of SRO214 thin-films grown on LSAT by pulsed laser deposition from a single crystal SRO327 target. The absence of superconductivity in films thinner than 50 nm is correlated with the in-plane misorientation mosaic twist, caused by in-plane screw dislocations, and with the most defective region near the SRO214/LSAT interface. The application of single crystal SRO327 targets offers a robust reliable platform for the creation of superconducting SRO214 thin-films and will initiate experimental studies involving multilayer structures and devices based on this highly important superconducting oxide.

Methods
Substrate preparation and growth. SRO214 films are grown by pulsed laser deposition onto \( 5 \times 5 \times 0.5 \text{ mm}^3 \) single crystal \((0\,0\,1)\) \((\text{LaAlO}_3)_{100}\left(\text{Sr}_2\text{AlTaO}_6)_{100}\right.\) (LSAT) with miscut angles of less than 0.05°. Prior to loading into the pulsed laser deposition chamber, the LSAT substrates are ultrasonicated for 10 min in acetone followed by 10 min in isopropanol, and subsequently dried using nitrogen gas. The LSAT substrates are attached to a 5C crystal \((10 \times 10 \times 3 \text{ mm}^3)\) with Pt paste (Tanaka Kikinzoku Kogyo K.K.) and secured with clips onto the substrate holder. The LSAT substrates are pre-baked for 30 min at 250°C in vacuum in the load-lock chamber. In the main chamber, the LSAT is annealed in ultra-high vacuum \((7.5 e^{-6} \text{ Pa})\) for 30 min at 950°C to promote terrace formation, with a warming ramp rate of 50°C min\(^{-1}\). The SRO214 films are grown in different oxygen pressures and KrF excimer laser (LPXpro 210 F Coherent Inc. 248 nm) energies as discussed in the main paper of SRO327 at a repetition rate of 2 Hz for the majority of the samples prepared in the same instrument, employing a Super-X detector with a total collection solid resolution polar Kerr effect measurements of SrRuO\(_4\) evidence for broken
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