Nickelate Superconductivity without Rare-Earth Magnetism: (La,Sr)NiO₂

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1. Introduction

The occurrence of unconventional superconductivity in cuprates has long motivated the search for manifestations in other layered transition metal oxides. Recently, superconductivity is found in infinite-layer nickelate (Nd,Sr)NiO₂ and (Pr,Sr)NiO₂ thin films, formed by topotactic reduction from the perovskite precursor phase. A topic of much current interest is whether rare-earth moments are essential for superconductivity in this system. In this study, it is found that with significant materials optimization, substantial portions of the La₁−ₓSrₓNiO₂ phase diagram can enter the regime of coherent low-temperature transport (x = 0.14 − 0.20), with subsequent superconducting transitions and a maximum onset of ≈9 K at x = 0.20. Additionally, the unexpected indication of a superconducting ground state in undoped LaNiO₂ is observed, which likely reflects the self-doped nature of the electronic structure. Combining the results of (La/Pr/Nd)₁−ₓSrₓNiO₂ reveals a generalized superconducting dome, characterized by systematic shifts in the unit cell volume and in the relative electron-hole populations across the lanthanides.
2. Results and Discussion

Figure 1a shows the temperature-dependent resistivity $\rho(T)$ for La$_{1-x}$Sr$_x$NiO$_2$ thin films ($x = 0$, 0.1, 0.2, and 0.3) from the initial study.\[2\] In all cases, the samples exhibited weak semiconducting behavior, with $x = 0.2$ showing the highest conductivity overall. The notion of increasing the electronic bandwidth, to further increase conductivity, motivated the subsequent investigation of (Nd,Sr)NiO$_2$ with the smaller cation Nd$^{3+}$ in place of La$^{3+}$. The undoped LaNiO$_2$ sample can be compared with other literature reports,\[24–27\] spanning almost two orders of magnitude in resistivity variation. While the resistivities of the initial samples were comparable to several of the prior reports,\[24,25\] others showed lower resistivity.\[26,27\] This indicated the possibility that the resistivities observed, and by extension for the doped samples, might not be intrinsic, but limited by materials imperfections. To explore this possibility, we worked to improve the crystallinity of (La,Sr)NiO$_2$ films following the approach recently taken for the Nd system \[12\] (Figures S1 and S2, Supporting Information), which led to pulsed laser deposition growth conditions at higher oxygen pressure, higher laser fluence, and smaller laser spot size (see Experimental Section for details). For comparison, the optimized $x = 0.2$ sample is also shown in Figure 1a, which shows substantially lower resistivity and metallic temperature dependence, followed by a superconducting transition.

This significant change in transport properties can be associated with changes in the crystallinity. In Figure 1b, we show x-ray diffraction (XRD) $\theta$–2$\theta$ wide scans for initial (non-superconducting; top) and optimized (superconducting; bottom) $x = 0.2$ samples, in which the two film peaks corresponding to (001)-oriented La$_{0.8}$Sr$_{0.2}$NiO$_2$ can be seen. First, in the optimized case, we observe a much more prominent 001 film peak, which has been found to be an important proxy for the crystallinity of the infinite layer phase.\[12\] Furthermore, as shown in Figure 1c, the full-width-at-half-maximum (FWHM) of rocking curve measurements on the 002 peaks of La$_{0.8}$Sr$_{0.2}$NiO$_2$ films grown via initial (red curve; top) and optimized (blue curve; bottom) conditions. d) Cross-sectional high-angle annular dark-field (HAADF) scanning transmission electron microscopy (STEM) image of the optimized La$_{0.8}$Sr$_{0.2}$NiO$_2$ film on SrTiO$_3$ substrate. e) Annular dark field (ADF) STEM image, simultaneously acquired elemental maps of the La-M$_{4,5}$, Sr-M$_{2,3}$ and Ti-L$_{2,3}$ edges, and false-colored composite image for which La, Sr, and Ti are shown in red, blue, and green, respectively. Scale bar indicates 1 nm.

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indicate atomically uniform films with no chemical segregation (Figure 1e). In contrast, Figure S3 (Supporting Information) shows cross-sectional STEM images of a La$_{0.8}$Sr$_{0.2}$NiO$_2$ film grown in the initial conditions,[2] displaying significantly higher disorder than an optimized sample (Figure 1d). Defects and extensive lattice disorder regularly extend throughout the entire depth of the film from surface to substrate interface (Figure S3b,c: Supporting Information). These microscopic observations further corroborate the significant enhancements of the crystallinity in the optimized (La,Sr)NiO$_2$ films.

Such materials improvements can be systematically extended to a wide range of compositions La$_{1-x}$Sr$_x$NiO$_2$ (0 ≤ $x$ ≤ 0.30). The formation of high-quality infinite-layer structure across all $x$ values studied here has been confirmed by XRD $\theta$–2$\theta$ scans (Figure S1a,c: Supporting Information). Figure 2 displays the temperature-dependent resistivity measured down to dilution refrigerator temperatures. At low and high doping levels ($x = 0.04, 0.06, 0.10, 0.24$, and 0.30), the resistivity curves show high-temperature metallic behavior, with an approximately logarithmic upturn at low temperatures,[28] analogous to that in the Nd/Pr systems.[13,14,18] Note that $x = 0.24$ and 0.30 show some evidence for decreasing crystallinity, with wider peaks in rocking curve measurements (Figure S1b,d: Supporting Information), likely reflecting the increasingly unstable high oxidation state of the perovskite precursor phase. Superconductivity emerges at intermediate doping ($0.14 \leq x \leq 0.20$), with a maximum superconducting transition temperature $T_c$ onset (defined as 90% of the 20 K resistivity, $T_c,90\%\rho$) of 9 K at $x = 0.20$.

Thus, we find that superconductivity in infinite-layer nickelates is not dependent on rare-earth states, namely, 4$f$ electrons and their corresponding (local) magnetic moments. Rather, the key observation is that with increased crystallinity, the resistivity is reduced, such that substantial portions of the (La,Sr)NiO$_2$ phase diagram are in the regime of coherent low-temperature transport, as indicated by the scale of the quantum sheet resistance $h/e^2$ per NiO$_2$ plane (black dots in Figure 2e). Furthermore, Figure S5 (Supporting Information) displays the suppression of superconductivity with perpendicular magnetic field up to 14 T, and the deduced in-plane Ginzburg-Landau coherence lengths for superconducting compositions, which are comparable to those found in Nd$_{1-x}$Sr$_x$NiO$_2$.[2,29]

Not only does the improved crystallinity reveal a superconducting dome, perhaps even more surprisingly, the optimized undoped LaNiO$_2$ is also in the coherent transport regime.
the closer lattice match between the precursor LaNiO$_3$ and SrTiO$_3$ substrate, and the improvements in growth. The second (3.38 Å) axis values. [30] At the diagonal defects, continued reduction.[25,34] For instance, an optimized LaNiO$_2$ thin film displays excellent crystallinity, largely free of the RP-2-axis orientation transition remains, but is suppressed with increasing crystallinity. This is presumably the remaining extended defect to be controlled for further improvements in materials quality, toward even cleaner LaNiO$_2$.

Finally, we discuss these optimized (La,Sr)NiO$_2$ results in the broader context of the doped infinite-layer nickelate films studied thus far. Together with the observation of superconductivity here, we can plot all existing experimental phase diagrams in Figure 4a. We note that while a small dip of $T_c$ is observed in the superconducting domes of La$_{1-x}$Sr$_x$NiO$_2$ and Nd$_{1-x}$Sr$_x$NiO$_3$, it is absent in Pr$_{1-x}$Sr$_x$NiO$_2$.[13,14,18] These results could suggest that a small distinction in the strain state may affect the ground state of the nickelates; for example, phases such as stripe order, widely observed in copper oxides[36,37] and subject to electronic correlations and the local chemical environment,[38] might be similarly present. Conversely, the strong disorder dependence of superconductivity observed here suggests that the detailed structure of the superconducting dome may yet reflect materials imperfections.

The variation in chemical composition impacts the interplanar spacing, which can be used to consolidate the different phase diagrams. In Figure 4b (and Figure S7, Supporting Information), we plot $T_{c,90\%}$ with respect to the measured c-axis lattice constant (in all cases, the in-plane lattice is clamped to the SrTiO$_3$ substrate). This is akin to assuming that only the unit cell dimensions are relevant for the variations observed. The resultant plot is highly suggestive of the existence of a generalized dome of superconductivity, across which different specific compounds cross. Note that this consideration neglects systematic trends in the electronic structure arising from Ni 3d – rare-earth 5d hybridization across the lanthanide series found in electronic structure calculations.[22,23,19] To examine this point, we have performed normal state Hall effect measurements for La$_{1-x}$Sr$_x$NiO$_2$ (0 ≤ x ≤ 0.30) (Figure 4c). Similar to Pr/Nd systems, the normal state Hall coefficient $R_H$ of La$_{1-x}$Sr$_x$NiO$_2$ increases with Sr substitution. Above intermediate doping levels ($x ≥ 0.14$), $R_H(T)$ monotonically increases with decreasing temperature. In particular, for samples with higher doping ($x ≥ 0.24$), $R_H(T)$ crosses zero in a consecutive manner.

Comparatively, in Figure 4d, we show $R_H(T)$ as a contour map across the available lanthanide series. In a simple two-band picture, this corresponds to a systematic shift in the relative compensation point between electron-hole populations (scaled by their mobilities) going from Nd to Pr and to La. These trends, however, are different from DFT-based calculations across the lanthanides that suggest the electron pockets grow from La to Pr/Nd.[23] One competing aspect is the effect of compressive strain, which tends to increase the electron pocket occupation and may dominate the evolution of the electronic structure.[22] La$_{1-x}$Sr$_x$NiO$_2$ is indeed more compressively strained on SrTiO$_3$ substrates than Pr$_{1-x}$Sr$_x$NiO$_2$ and Nd$_{1-x}$Sr$_x$NiO$_2$. Further investigation of these competing tendencies may lead to an understanding of the underlying structure of the superconducting...
dome (Figure 4b), and the absence of superconductivity in bulk samples.[40,41]

3. Conclusion

Significant enhancements of the crystallinity and electrical conductivity were achieved in (La,Sr)NiO$_2$, associated with optimization of both growth and reduction conditions. In all cases where the low-temperature normal state resistivity falls below a resistance quantum per NiO$_2$ plane, a superconducting transition is observed. This results in a doping-dependent superconducting dome that is somewhat smaller than those found in Nd$_{1-x}$Sr$_x$NiO$_2$ and Pr$_{1-x}$Sr$_x$NiO$_2$. These trends are mirrored in systematic shifts in the normal state Hall effect and unit cell volume across the lanthanides. In addition, we observe indications of superconductivity in undoped LaNiO$_2$, which has not been previously observed in any of the undoped infinite-layer nickelates. Taken together, these results indicate that rare-earth magnetism is not essential for superconductivity, and rather that disorder plays a key role in delineating between a superconducting or weakly-insulating ground state in this system.

4. Experimental Section

Thin Film Growth: Solid-solution La$_{1-x}$Sr$_x$NiO$_3$ nickelate thin films (5.5–10 nm thick) were grown on single-crystalline SrTiO$_3$ (001)
substrates by pulsed laser deposition using stoichiometric polycrystalline targets ablated with a KrF excimer laser (wavelength = 248 nm), followed in some cases by the growth of a ≈ 2 nm SrTiO3 capping layer. Prior to the growth, SrTiO3 substrates were pre-annealed at 930 °C under an oxygen partial pressure of 5 × 10⁻⁶ Torr, in order to obtain an atomically flat surface topography. During growth, the substrate temperature was kept at 570 °C. In the initial growth conditions, La1−xSr xNiO3 thin films were grown under oxygen pressure of 35 mTorr using the laser fluence of 1.47 J cm⁻² (laser spot area 3.9 mm²); after optimization, the growth conditions were a laser fluence of 1.39 and 2.19 J cm⁻² for undoped LaNiO3 and doped La1−xSr xNiO3 (x ≠ 0), respectively (laser spot area 2.6 mm²). The oxygen pressure during the growth was 200 and 250 mTorr for undoped LaNiO3 and doped La1−xSr xNiO3 (x ≠ 0), respectively. For SrTiO3 capping layer growth, a laser fluence of 0.87 J cm⁻² and oxygen pressure of 200 mTorr were used. The laser repetition was 4 Hz for all cases.

Topotactic Reduction: In order to obtain films of the infinite-layer nickelate La1−xSr xNiO2, the as-grown precursor perovskite thin films were loosely wrapped with aluminum foil and placed in Pyrex glass tubes with calcium hydride (CaH2) powder (≈ 0.1 g). The glass tubes were evacuated to below 0.1 mTorr using a rotary pump and subsequently sealed using a hydrogen torch. The sealed tube was then heated in a tube furnace at 260 °C for 60 min for SrTiO3 capped films and at 240 °C for 60 min for uncapped films unless otherwise specified (e.g., the reduction study of Figure S2, Supporting Information). The thermal ramping and cooling rates were 10 °C min⁻¹.

Thin Film Characterization: The X-ray diffraction (XRD) θ-2θ profiles and rocking curves were collected using a monochromatic Cu Kα radiation source. The rocking curve measurements were performed for the 002-diffraction peaks of La1−xSr xNiO2. To compare the sample quality, the intensities were normalized. The longitudinal and Hall resistivity of the 2.5 mm × 5 mm films were measured using a standard six-point geometry with Al wire bonded contacts. Normal state Hall coefficients were obtained from the Hall resistivity ρxy, which showed linear magnetic field dependence up to 9 T.

Scanning Transmission Electron Microscopy and Analysis: Cross-sectional scanning transmission electron microscopy (STEM) specimens were prepared using a standard focused ion beam (FIB) lift-out process on a Thermo Scientific Helios G4 UX FIB. Samples were imaged on an aberration-corrected FEI Titan Themis microscope operated at 300 keV with a 30 mrad probe-forming convergence angle and a 68 mrad inner collection angle. Electron energy-loss spectroscopy (EELS) was performed on the same Titan Themis system equipped with a 965 GIF Quantum ER and a Gatan K2 Summit direct electron detector operated in electron counting mode. Analysis of local lattice orientation and spacing was performed using the local wave-fitting method described in ref. [35] with a Fourier mask size corresponding to 2 nm real-space resolution.

Note: During the preparation of this manuscript, we became aware of a report of superconductivity in (La,Ca)NiO2.[42]

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

nickelates, rare-earth, superconductivity, thin films

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