LETTER

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Magnetic-field-induced charge-stripe order in the high-temperature superconductor $YBa_2Cu_3O_y$

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Electronic charges introduced in copper-oxide (CuO_2) planes generate high-transition-temperature (T_c) superconductivity but, under special circumstances, they can also order into filaments called stripes[1]. Whether an underlying tendency towards charge order is present in all copper oxides and whether this has any relationship with superconductivity are, however, two highly controversial issues[2,3]. To uncover underlying electronic order, magnetic fields strong enough to destabilize superconductivity can be used. Such experiments, including quantum oscillations[4–6] in $YBa_2Cu_3O_y$ (an extremely clean copper oxide in which charge order has not until now been observed) have suggested that superconductivity competes with spin, rather than charge, order[7–9]. Here we report nuclear magnetic resonance measurements showing that high magnetic fields actually induce charge order, without spin order, in the CuO_2 planes of $YBa_2Cu_3O_y$. The observed static, unidirectional, modulation of the charge density breaks translational symmetry, thus explaining quantum oscillation results, and we argue that it is most probably the same 4*a*-periodic modulation as in stripe-ordered copper oxides[1]. That it develops only when superconductivity fades away and near the same 1/8 hole doping as in $La_{2-x}Ba_xCuO_4$ (ref. 1) suggests that charge order, although visibly pinned by CuO chains in $YBa_2Cu_3O_y$, is an intrinsic propensity of the superconducting planes of high-T_c copper oxides.

The ortho II structure of $YBa_2Cu_3O_{6.54}$ ($p = 0.108$, where p is the hole concentration per planar Cu) leads to two distinct planar Cu NMR sites: Cu2F are those Cu atoms located below oxygen-filled chains, and Cu2E are those below oxygen-empty chains[10]. The main discovery of our work is that, on cooling in a field H_0 of 28.5 T along the c axis (that is, in the conditions for which quantum oscillations are resolved; see Supplementary Materials), the Cu2F lines undergo a profound change, whereas the Cu2E lines do not (Fig. 1). To first order, this change can be described as a splitting of Cu2F into two sites having both different hyperfine shifts $K = \langle h_z \rangle / H_0$ (where $\langle h_z \rangle$ is the hyperfine field due to electronic spins) and quadrupole frequencies ν_Q (related to the electric field gradient). Additional effects might be present (Fig. 1), but they are minor in comparison with the observed splitting. Changes in field-dependent and temperature-dependent orbital occupancy (for example $d_{x^2-y^2}$ versus $d_{z^2-r^2}$) without on-site change in electronic density are implausible, and any change in out-of-plane charge density or lattice would affect Cu2E sites as well. Thus, the change in ν_Q can only arise from a differentiation in the charge density between Cu2F sites (or at the oxygen sites bridging them). A change in the asymmetry parameter and/or in the direction of the principal axis of the electric field gradient could also be associated with this charge differentiation, but these are relatively small effects.

The charge differentiation occurs below $T_{charge} = 50 \pm 10$ K for $p = 0.108$ (Fig. 1 and Supplementary Figs 9 and 10) and 67 ± 5 K for $p = 0.12$ (Supplementary Figs 7 and 8). Within error bars, for each of the samples T_{charge} coincides with T_0, the temperature at which the Hall constant R_H becomes negative, an indication of the Fermi surface reconstruction[11–13]. Thus, whatever the precise profile of the static charge modulation is, the reconstruction must be related to the translational symmetry breaking by the charge ordered state.

The absence of any splitting or broadening of Cu2E lines implies a one-dimensional character of the modulation within the planes and imposes strong constraints on the charge pattern. Actually, only two types of modulation are compatible with a Cu2F splitting (Fig. 2). The first is a commensurate short-range (2*a* or 4*a* period) modulation running along the (chain) b axis. However, this hypothesis is highly unlikely: to the best of our knowledge, no such modulation has ever been observed in the CuO_2 planes of any copper oxide; it would therefore have to be triggered by a charge modulation pre-existing in the filled chains. A charge-density wave is unlikely because the finite-size chains are at best poorly conducting in the temperature and doping range discussed here[11,14]. Any inhomogeneous charge distribution such as Friedel oscillations around chain defects would broaden rather than split the lines. Furthermore, we can conclude that charge order occurs only for high fields perpendicular to the planes because the NMR lines neither split at 15 T nor split in a field of 28.5 T parallel to the CuO_2 planes (along either a or b), two situations in which superconductivity remains robust (Fig. 1). This clear competition between charge order and superconductivity is also a strong indication that the charge ordering instability arises from the planes.

The only other pattern compatible with NMR data is an alternation of more and less charged Cu2F rows defining a modulation with a period of four lattice spacings along the a axis (Fig. 2). Strikingly, this corresponds to the (site-centred) charge stripes found in $La_{2-x}Ba_xCuO_4$ at doping levels near $p = x = 0.125$ (ref. 1). Being a proven electronic instability of the planes, which is detrimental to superconductivity[2], stripe order not only provides a simple explanation of the NMR splitting but also rationalizes the striking effect of the field. Stripe order is also fully consistent with the remarkable similarity of transport data in $YBa_2Cu_3O_y$ and in stripe-ordered copper oxides (particularly the dome-shaped dependence of T_0 around $p = 0.12$)[11–13]. However, stripes must be parallel from plane to plane in $YBa_2Cu_3O_y$, whereas they are perpendicular in, for example, $La_{2-x}Ba_xCuO_4$. We speculate that this explains why the charge transport along the c axis in $YBa_2Cu_3O_y$ becomes coherent in high fields below T_0 (ref. 15). If so, stripe fluctuations must be involved in the incoherence along c above T_0.

Once we know the doping dependence of ν_Q (ref. 16), the difference $\Delta\nu_Q = 320 \pm 50$ kHz for $p = 0.108$ implies a charge density variation as small as $\Delta p = 0.03 \pm 0.01$ hole between Cu2Fa and Cu2Fb. A canonical stripe description ($\Delta p = 0.5$ hole) is therefore inadequate at the NMR timescale of $\sim 10^{-5}$ s, at which most (below T_0) or all (above T_0) of the charge differentiation is averaged out by fluctuations faster than $10^5\ s^{-1}$. This should not be a surprise: the metallic nature of the compound at all fields is incompatible with full charge order, even if this order is restricted to the direction perpendicular to the stripes[17]. Actually, there is compelling evidence of stripe fluctuations down to very low temperatures in stripe-ordered copper oxides[18], and indirect

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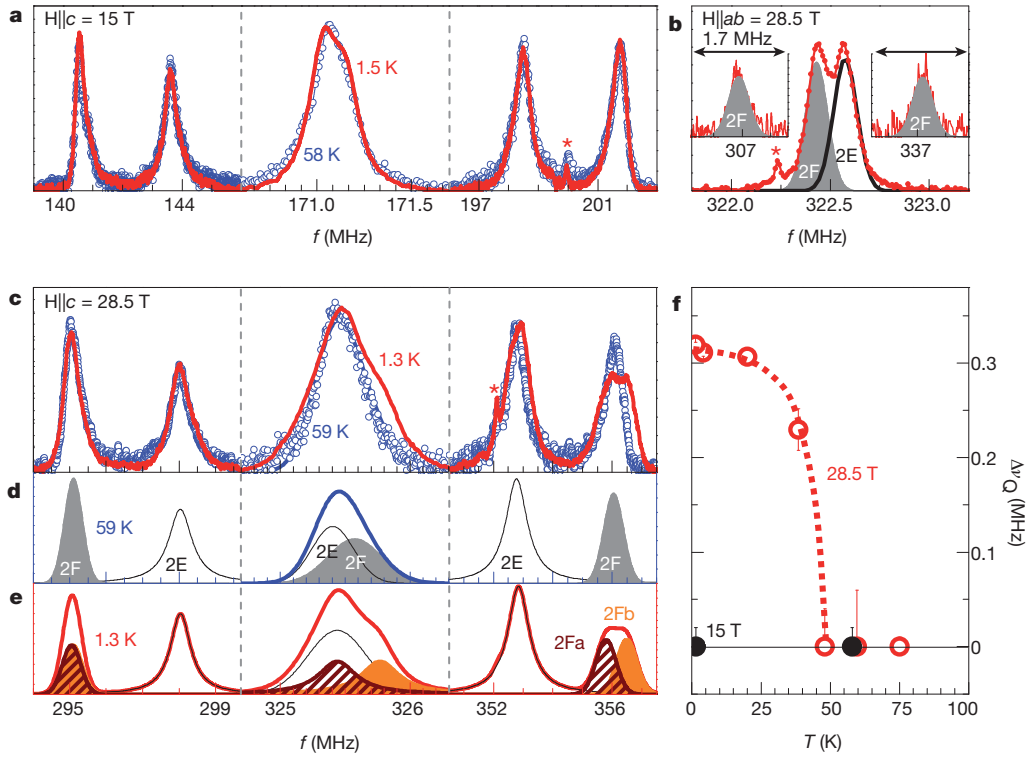

Figure 1 | High-field NMR spectra of $YBa_2Cu_3O_{6.54}$ (ortho II, $p = 0.108$). **a**, ^{63}Cu(2) NMR lines for $H_0||c$ (15 T) do not show any temperature-induced splitting. A small overall shift has been subtracted for clarity. The slight change in shape of the central line at 1.5 K is due to Cu2F broadening and T_2 shortening, but the Cu2E/Cu2F positions are unchanged with respect to higher temperatures and lower fields. A small background on the central line at 58 K has been subtracted (see Supplementary Information). The difference in ν_Q between Cu2E and Cu2F is due to the presence of empty and filled chains creating different local environments, minute differences in the orbital occupations and, possibly, slightly different charge densities at these sites (this last possibility is as yet unsettled, but this is not directly relevant to the present work because it is field independent up to at least 300 K). **b**, Cu2F lines for $H_0||ab$ (28.5 T) show no splitting or broadening at 1.5 K. Cu2E satellites are not shown. **c**, ^{63}Cu lines for $H_0||c$ (28.5 T). **d**, **e**, Decomposition of the spectrum into the different sites above T_{charge} (**d**) and below T_{charge} (**e**). The charge density modulation below $T_{charge} \approx 50$ K causes Cu2F to split into Cu2Fa and Cu2Fb (higher-density site for its larger ν_Q value[16]). A similar NMR splitting might have been observed in stripe-ordered nickelates[30]. The parameters are given in Supplementary Table 1. The central line positions are determined from the positions of the satellites, and they are independently confirmed in Supplementary Figs 4 and 5. Asterisks indicate signals from ^{63}Cu in oxygen-empty chains. A subtle bump on the low-frequency side of both Cu2F and Cu2E high-frequency satellites is not understood, but is a minor effect compared with the splitting of the Cu2F site and it could be either intrinsic (weak additional magnetic or charge modulation) or extrinsic (defects in the ordered pattern). **f**, Quadrupolar contribution to the splitting of the high-frequency Cu2F satellite (see Supplementary Fig. 9 for details). Error bars include the statistical error from the fit and the uncertainty on the presence of a splitting at high temperatures.

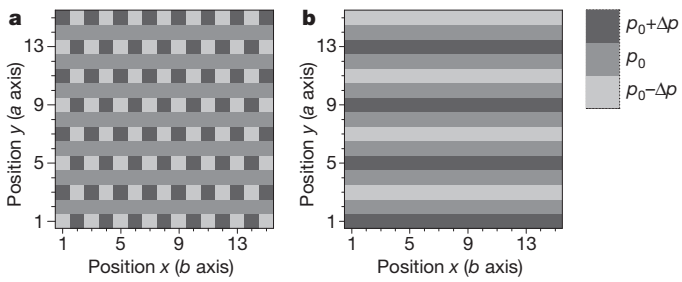

Figure 2 | Charge density modulations compatible with NMR spectra. **a**, $2b$-periodic charge modulation along the chain (***b***) axis: $p(x,y) = p_0 + p_1 \sin(\pi y/2)\cos(\pi x)$ with $p_0 = 0.108$ being the mean hole content and $\Delta p = 2p_1 = 0.03$ the amplitude of the modulation. Cu2F sites lie at odd positions on the a axis, and Cu2E sites at even positions. A similar $4b$-period modulation of the form $p(x,y) = p_0 + p_1\sqrt{2}\sin(\pi y/2)\cos(\pi x/2 + \pi/4)$ is also possible. These two modulations, though consistent with the NMR spectra, are unable to explain the field dependence of the modulation as well as the correlation between our NMR data, transport measurements and '1/8 anomalies'. **b**, Stripe order with period $4a$ perpendicular to the chain (b) axis: $p(x,y) = p_0 + p_1 \sin(\pi y/2)$. To minimize Coulomb repulsion, the doped holes tend to align below the neutral (filled) chains rather than below the Cu^+ (empty) chains, explaining why the higher charge density sites are the Cu2F rather than the 2E sites. Note that, in either case, an additional transverse modulation $p(y) = p_2 \cos(\pi y)$ resulting from the ortho II potential and present at any temperature and field cannot be excluded. In the more complex ortho VIII structure of the $p = 0.12$ sample, it is also possible to obtain the same striped charge pattern with the high density sites only below filled chains.

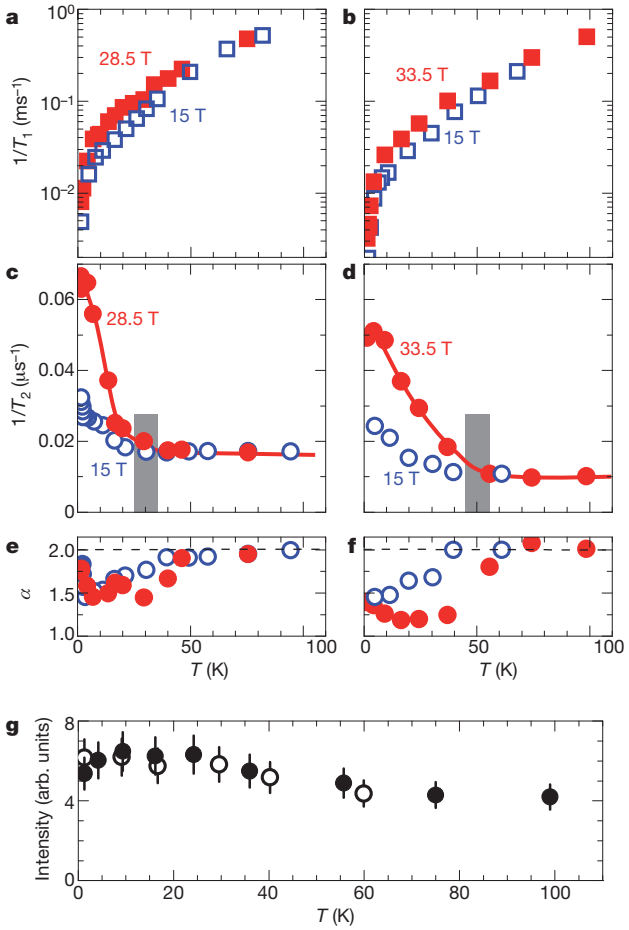

Figure 3 | Slow spin fluctuations instead of spin order. **a**, **b**, Temperature dependence of the planar ^{63}Cu spin-lattice relaxation rate $1/T_1$ for $p = 0.108$ (**a**) and $p = 0.12$ (**b**). The absence of any peak/enhancement on cooling rules out the occurrence of a magnetic transition. **c**, **d**, Increase in the ^{63}Cu spin–spin relaxation rate $1/T_2$ on cooling below $\sim T_{charge}$, obtained from a fit of the spin-echo decay to a stretched form $s(t) \propto \exp(-(t/T_2)^{\alpha})$, for $p = 0.108$ (**c**) and $p = 0.12$ (**d**). **e**, **f**, Stretching exponent α for $p = 0.108$ (**e**) and $p = 0.12$ (**f**). The deviation from $\alpha = 2$ on cooling arises mostly from an intrinsic combination of Gaussian and exponential decays, combined with some spatial distribution of T_2 values (Supplementary Information). The grey areas define the crossover temperature T_{slow} below which slow spin fluctuations cause $1/T_2$ to increase and to become field dependent; note that the change of shape of the spin-echo decay occurs at a slightly higher ($\sim$+15 K) temperature than T_{slow}. T_{slow} is slightly lower than T_{charge}, which is consistent with the slow fluctuations being a consequence of charge-stripe order. The increase of α at the lowest temperatures probably signifies that the condition $\gamma\langle h_z^2\rangle^{1/2}\tau_c \ll 1$, where τ_c is the correlation time, is no longer fulfilled, so that the associated decay is no longer a pure exponential. We note that the upturn of $1/T_2$ is already present at 15 T, whereas no line splitting is detected at this field. The field therefore affects the spin fluctuations quantitatively but not qualitatively. **g**, Plot of NMR signal intensity (corrected for a temperature factor $1/T$ and for the T_2 decay) against temperature. Open circles, $p = 0.108$ (28.5 T); filled circles, $p = 0.12$ (33.5 T). The absence of any intensity loss at low temperatures also rules out the presence of magnetic order with any significant moment. Error bars represent the added uncertainties in signal analysis, experimental conditions and T_2 measurements. All measurements are with $H||c$.

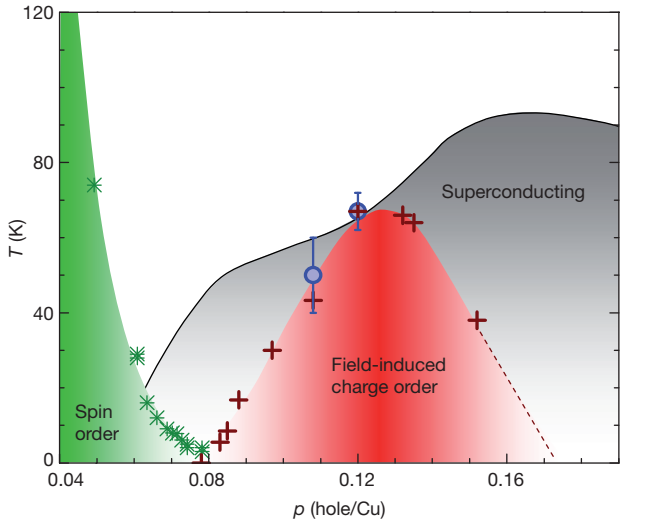

Figure 4 | Phase diagram of underdoped $YBa_2Cu_3O_y$. The charge ordering temperature T_{charge} (defined as the onset of the Cu2F line splitting; blue open circles) coincides with T_0 (brown plus signs), the temperature at which the Hall constant R_H changes its sign. T_0 is considered as the onset of the Fermi surface reconstruction[11–13]. The continuous line represents the superconducting transition temperature T_c. The dashed line indicates the speculative nature of the extrapolation of the field-induced charge order. The magnetic transition temperatures (T_{spin}) are from muon-spin-rotation (μSR) data (green stars)[27]. T_0 and T_{spin} vanish close to the same critical concentration $p = 0.08$. A scenario of field-induced spin order has been predicted for $p > 0.08$ (ref. 8) by analogy with $La_{1.855}Sr_{0.145}CuO_4$, for which the non-magnetic ground state switches to antiferromagnetic order in fields greater than a few teslas (ref. 7 and references therein). Our work, however, shows that spin order does not occur up to $\sim$30 T. In contrast, the field-induced charge order reported here raises the question of whether a similar field-dependent charge order actually underlies the field dependence of the spin order in $La_{2-x}Sr_xCuO_4$ and $YBa_2Cu_3O_{6.45}$. Error bars represent the uncertainty in defining the onset of the NMR line splitting (Fig. 1f and Supplementary Figs 8–10).

evidence (explaining the rotational symmetry breaking) over a broad temperature range in $YBa_2Cu_3O_y$ (refs 14, 19–22). Therefore, instead of being a defining property of the ordered state, the small amplitude of the charge differentiation is more likely to be a consequence of stripe order (the smectic phase of an electronic liquid crystal[17]) remaining partly fluctuating (that is, nematic).

In stripe copper oxides, charge order at $T = T_{charge}$ is always accompanied by spin order at $T_{spin} < T_{charge}$. Slowing down of the spin fluctuations strongly enhances the spin–lattice ($1/T_1$) and spin–spin ($1/T_2$) relaxation rates between T_{charge} and T_{spin} for ^{139}La nuclei. For the more strongly hyperfine-coupled ^{63}Cu, the relaxation rates become so large that the Cu signal is gradually 'wiped out' on cooling below T_{charge} (refs 18, 23, 24). In contrast, the ^{63}Cu(2) signal here in $YBa_2Cu_3O_y$ does not experience any intensity loss and $1/T_1$ does not show any peak or enhancement as a function of temperature (Fig. 3). Moreover, the anisotropy of the linewidth (Supplementary Information) indicates that the spins, although staggered, align mostly along the field (that is, c axis) direction, and the typical width of the central lines at base temperature sets an upper magnitude for the static spin polarization as small as $g\langle S_z\rangle \leq 2 \times 10^{-3}\mu_B$ for both samples in fields of $\sim$30 T. These consistent observations rule out the presence of magnetic order, in agreement with an earlier suggestion based on the presence of free-electron-like Zeeman splitting[6].

In stripe-ordered copper oxides, the strong increase of $1/T_2$ on cooling below T_{charge} is accompanied by a crossover of the time decay of the spin-echo from the high-temperature Gaussian form $\exp(-\frac{1}{2}(t/T_{2G})^2)$ to an exponential form $\exp(-t/T_{2E})$[18,23]. A similar crossover occurs here, albeit in a less extreme manner because of the absence of magnetic order: $1/T_2$ sharply increases below T_{charge} and the decay actually becomes a combination of exponential and Gaussian decays (Fig. 3). In Supplementary Information we provide evidence that the typical values of the $1/T_{2E}$ below T_{charge} imply that antiferromagnetic (or 'spin-density-wave') fluctuations are slow enough to appear frozen on the timescale of a cyclotron orbit $1/\omega_c \approx 10^{-12}$ s. In principle, such slow fluctuations could reconstruct the Fermi surface, provided that spins are correlated over large enough distances[25,26] (see also ref. 9). It is unclear whether this condition is fulfilled here. The

fluctuations could also appear frozen on the timescale of an elastic neutron scattering experiment, as in $YBa_2Cu_3O_{6.45}$ ($p \approx 0.08$)[8,19]. However, there is a fundamental difference between $p = 0.08$ and the $p = 0.108$–0.12 samples here, which is not a question of experimental timescale: our NMR data in $YBa_2Cu_3O_{6.45}$ (T. Wu, H. Mayaffre, S. Krämer, M. Horvatić, C. Berthier, C. T. Lin, D. Haug, T. Loew, V. Hinkov, B. Keimer and M.-H. Julien, unpublished observations) are completely different from those reported here, with unequivocal evidence of spin order. Even if we cannot exclude the possibility that a freezing at the NMR timescale occurs at much lower temperatures and/or higher field, spin order seems to be absent over a range of temperatures and fields where charge order and quantum oscillations are observed, indicating that it cannot be an essential ingredient of these two phenomena. Actually, the phase diagram of underdoped $YBa_2Cu_3O_y$ (Fig. 4) even suggests that antiferromagnetic order[27] and Fermi surface reconstruction around $p = 0.12$ (refs 4–6, 11–13) are mutually exclusive phenomena. It is tempting to associate the absence of spin order to the remaining stripe fluctuations discussed above.

The implications of our results go beyond the microscopic explanation of quantum oscillation experiments. Although it is the chain structure that manifestly pins stripe order in $YBa_2Cu_3O_y$, the chains should not be taken as being responsible for the whole stripe phenomenon here. First, charge ordering has an onset near T_0, where the Hall effect changes its sign, and this sign change has been shown to be the same planar '1/8 anomaly' as in stripe-ordered copper oxides[12,13]. Second, T_0 (ref. 12 and Fig. 4) is a continuous function of hole doping, irrespective of the oxygen ordering sequence. In particular, the highest T_{charge} is found for the ortho VIII structure ($p = 0.12$), which is more complex and of shorter range than ortho II ($p \sim 0.1$). Therefore, neither the presence of chains in $YBa_2Cu_3O_y$ nor the strong disorder typical of La-based stripe copper oxides can be the sole origin of charge stripes in ultra-clean $YBa_2Cu_3O_y$. Stripe correlations originating from the superconducting planes are consistent with the field-tuned competition between charge order and superconductivity revealed here. Our observation of unidirectional charge order in $YBa_2Cu_3O_y$ ($p = 0.11$–0.12) thus strengthens the idea that there is an intrinsic and most probably ubiquitous[28,29] propensity towards charge ordering in high-T_c copper oxides that is most apparent around $p = 1/8$.

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Supplementary Information is linked to the online version of the paper at www.nature.com/nature.

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