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# Temperature evolution of the magnetic excitations in charge ordered La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub>

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#### Abstract

Polarized- and unpolarized-neutron scattering was used to study the temperature evolution of the magnetic excitations of charge ordered  $La_{5/3}Sr_{1/3}NiO_4$ . We studied two features in detail: (i) the resonance-like scattering at 27 meV in the quasi-two-dimensional magnetic excitations from the ordered Ni<sup>2+</sup> (S = 1) spins, and (ii) the diffuse scattering associated with quasi-one-dimensional antiferromagnetic correlations along the stripes. Although both these features persist up to at least ~125 K, their temperature dependences are found to be quite different. We argue from the results that the resonance-like feature observed in  $La_{2-x}Sr_xNiO_4$  ( $x \sim 1/3$ ) is not caused by a coupling between the spin dynamics of the two magnetic sub-systems.

(Some figures in this article are in colour only in the electronic version)

# 1. Introduction

The role of stripes in the copper oxide superconductors has been discussed in great detail ever since the discovery of spin–charge stripe order in non-superconducting  $La_{1.48}Nd_{0.4}Sr_{0.12}CuO_4$  [1]. An interesting problem still under debate concerns the spin dynamics, specifically whether the existence of stripe correlations can be inferred from the spin excitation spectrum. One approach to this problem has been to compare the magnetic excitation spectrum in the hole-doped cuprate superconductors, which has recently been studied over a wide energy range [2–8], with the excitations in other, nonsuperconducting, oxides exhibiting similar stripe order, especially the layered nickelates  $La_{2-x}Sr_xNiO_{4+\delta}$  (LSNO) [9–12]. An understanding of the similarities and differences may point the way to elucidating the mechanism of high-temperature cuprate superconductivity.

A notable feature in the spin excitation spectrum of the cuprates is the so-called 'resonance peak'. This is a peak observed by magnetic neutron scattering at temperatures below the superconducting transition temperature  $T_c$ , which in some cases also persists above  $T_c$ . It was first observed in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$ </sub> (YBCO) [13], but similar features have since been observed in La<sub>2-x</sub>Sr<sub>x</sub>CuO<sub>4</sub> (LSCO) [6], Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8+ $\delta$ </sub> [14], Tl<sub>2</sub>Ba<sub>2</sub>CuO<sub>6+ $\delta$ </sub> [15] and even in the electron-doped superconductors Pr<sub>0.88</sub>LaCe<sub>0.12</sub>CuO<sub>4- $\delta$ </sub> [16] and Nd<sub>1.85</sub>Ce<sub>0.15</sub>CuO<sub>4- $\delta$ </sub> [17]. The resonance peak is localized in both wavevector and energy. In YBCO the peak appears at the antiferromagnetic wavevector [18, 19]<sup>4</sup>, which for a square lattice is **Q**<sub>AF</sub> = (0.5, 0.5) in reciprocal lattice units, whereas in LSCO the peak is at an incommensurate wavevector. The resonance energy tends to scale with  $T_c$  [16, 20].

Interestingly, a resonance-like feature has also been observed in the spin excitation spectrum of stripe-ordered LSNO for doping levels  $x \sim 1/3$  [9, 11, 12]. The 'resonance' in LSNO cannot be explained within the framework of linear spin-wave theory based on the accepted magnetic ordering

 $<sup>^4\,</sup>$  In YBCO there are actually two resonance peaks, corresponding to in-phase or out-of-phase spin fluctuation modes on adjacent CuO<sub>2</sub> layers within the bilayer [18, 19].

pattern of the  $Ni^{2+}$  spins. A number of other explanations for its origin have been discussed [9, 12], but so far no theoretical analysis has been performed to test any of these other possibilities. The aim of the experiments described here was to obtain more clues about the physical origin of the resonance in LSNO. The ultimate objective is to establish whether or not there is any possible link between the unexplained feature observed in the spin dynamics in LSNO and the resonance peak in the cuprate superconductors.

### 2. Magnetic excitations in $La_{2-x}Sr_xNiO_{4+\delta}$

The charge and magnetic ordering properties of  $La_{2-x}Sr_xNi$  $O_{4+\delta}$  have been studied in detail by diffraction techniques [21–24]. For x = 1/3,  $\delta = 0$ , holes doped into the NiO<sub>2</sub> layers segregate below  $T_{\rm CO} \approx 240$  K into stripes running at 45° to the Ni-O bonds. Antiferromagnetic ordering of the spins in the hole-poor regions between the charge stripes occurs below  $T_{\rm m} \approx 200$  K, i.e. slightly lower than  $T_{\rm CO}$ . In general, the periodicities of the magnetic order and charge order are different. The periodicities depend on the hole concentration  $n_{\rm h} = x + 2\delta$  and are also influenced by competition between the spin and charge interactions [23]. The composition  $x = 1/3, \delta = 0$  ( $n_{\rm h} = 1/3$ ) studied here is special because (i) the spin and charge order have the same period [24], and (ii) the stripe pattern at  $n_{\rm h} = 1/3$  is commensurate with the lattice. The spin and charge commensurability in this composition makes the stripe order particularly stable [23, 24].

The assumed spin and charge ordering within the tetragonal *ab* plane of  $La_{5/3}Sr_{1/3}NiO_4$  is shown in figure 1(a). AFM ordered spins are located on the Ni<sup>2+</sup> sites and holes are shown located on Ni<sup>3+</sup> (site-centred stripes). Another possibility is that the holes are located in the Ni–O bonds (bond-centred stripes). Either way, the charge stripes act as antiphase boundaries for the AFM order. There are also spin degrees of freedom associated with the charge stripes but they do not order.

The spin dynamics of La5/3Sr1/3NiO4 has been studied throughout the Brillouin zone by neutron spectroscopy [9, 12, 25]. The spectrum comprises two components: (1) strongly dispersive, two-dimensional (2D), spin-wave-like excitations associated with the magnetic ordering wavevector (1/3, 1/3, 0) and equivalent positions (see figure 1(b)); (2) dynamic quasi-one-dimensional (q-1D) spin correlations along the charge stripes which produce diffuse ridges of inelastic scattering running perpendicular to the stripes. Neither component exhibits any dispersion along the  $c^*$  direction in the energy range studied, so correlations between the layers can be neglected. The spectrum associated with the magnetic order extends up to 80 meV and for the most part can be described very well by a linear spin-wave model based on a nearest-neighbour Heisenberg spin Hamiltonian with an easyplane anisotropy. A fit to this model yields  $J'/J \approx 0.5$ , where J' and J are the inter-stripe and intra-stripe exchange couplings between nearest-neighbour Ni<sup>2+</sup> spins (figure 1(a)). Hence, the magnetic coupling across the stripes is comparable to that within the stripes. The q-1D inelastic scattering, which derives predominantly from c-axis spin components, exhibits



Figure 1. (a) Spin arrangement on the Ni sites in an *ab* plane of  $La_{5/3}Sr_{1/3}NiO_4$ . Broken lines indicate the charge stripes and J, J' indicate the intra-and inter-stripe exchange interactions, respectively. (b) The location in reciprocal space of the stripe superlattice Bragg reflections and diffuse scattering from La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub>. The charge stripe configuration in (a) leads to Bragg reflections along the [110] direction (black circles). The orthogonal charge stripe domain causes magnetic Bragg reflections along the  $[1\overline{10}]$  direction (red circles with a black outline). In our measurements the two domains are found to be equally populated [9]. Solid black and broken red lines indicate the lines along which inelastic diffuse scattering is observed from the dynamic quasi-one-dimensional (q-1D) AFM spin correlations of the charge stripe electrons [25]. Solid black lines indicate diffuse scattering arising from the [110] stripe domain shown in (a), and broken red lines indicate diffuse scattering arising from the orthogonal [110] stripe domain. (c) Polarized neutron scattering measurement of part of the magnetic excitation spectrum of  $La_{5/3}Sr_{1/3}NiO_4$  showing the resonance-like peak at  $\sim 27$  meV. The data, which are reproduced from [9], show an energy scan with the neutron scattering vector fixed at a magnetic zone centre. The signal is purely magnetic since the scattering is measured in the spin-flip channel with the neutron polarization **P** parallel to the scattering vector Q.

a sinusoidal dispersion which extends up to 10 meV and is assumed to be the lower bound of the two-spinon continuum.

The feature of interest for the present study lies between 10 and 30 meV. In this energy range the spin excitation spectrum deviates markedly from the spin-wave model. This part of the spectrum is reproduced in figure 1(c), which shows polarized neutron scattering as a function of energy at a fixed wavevector  $\mathbf{Q} = (4/3, 4/3, 0)$ . The reduction in intensity below 7 meV is a gap caused by the quenching of out-of-plane spin fluctuations due to the easy-plane anisotropy. This is

clearly established from polarized-neutron measurements [11]. Between 10 and 30 meV, however, the form of the scattering is not at all understood. The magnetic structure shown in figure 1(a) can be described as a two-sublattice AFM, and according to linear spin-wave theory the scattering cross-section should decrease monotonically with energy for all energies above the spin gap. What is observed in practice, however, is an initial reduction in intensity to a minimum near 15 meV followed by an increase in intensity culminating in a peak centred near 27 meV. This latter peak is the resonance-like feature central to this paper.

A natural question to ask is whether this resonance-like excitation in the spin excitations from the AFM ordered spins could arise from coupling to the q-1D spin correlations. Any such coupling is frustrated at low energies because the stripes are at a centre of symmetry of the magnetic ordering pattern (figure 1(a)). It is conceivable, however, that effects due to coupling might appear at higher energies. The idea of the present work was to test this possibility by comparing the temperature dependence of the scattering in the range 10–30 meV with that of the q-1D scattering. We find that whereas the anomalous peak persists up to at least 125 K, the q-1D scattering has all but disappeared by 125 K. This implies that the 'resonance peak' is not caused by coupling between the two spin systems.

### 3. Experimental details

A single crystal of  $La_{5/3}Sr_{1/3}NiO_{4+\delta}$  was used for the experiments. The crystal was a rod of 7-8 mm diameter and  $\sim$ 40 mm in length. The sample was grown from high-purity powders by the floating-zone technique [26]. The oxygen content was determined by thermogravimetric analysis to be  $\delta = 0.01 \pm 0.01$ . Henceforth we will assume a stoichiometric oxygen content. Electron probe microscopy on single crystals of  $La_{2-x}Sr_xNiO_{4+\delta}$  grown by the floating-zone technique have shown that the cation ratios agree with the nominal values [27]. This finding is consistent with our observation that the stripe period in our crystal of La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub> is equal to three lattice spacings to within better than 0.5%, as expected for a hole content  $n_{\rm h} = 1/3$ . Diffraction measurements [28] have shown that for  $x > 0.2 \text{ La}_{2-x} \text{Sr}_x \text{NiO}_4$  adopts the I4/mmm space group, which has one La(Sr) site. It is assumed that the Sr ions are randomly distributed among the La sites; there is no evidence in diffraction experiments for the existence of Sr ordering or clustering.

The crystal used in this work was also used in some of our previous investigations of the magnetic excitation spectrum of La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub> [9, 11, 12, 25]. Here, as previously, we index reciprocal space with respect to the body-centred tetragonal unit cell of the *I*4/*mmm* lattice, with unit cell parameters  $a \approx$  3.8 Å,  $c \approx$  12.7 Å. Although the magnetic excitation spectrum is essentially 2D in character, we give for completeness all three components (*hkl*) of the neutron scattering vector for the experimental measurements.

The polarized-neutron experiments were performed on the triple-axis spectrometer IN20 at the Institut Laue-Langevin. The energies of the incident and scattered neutrons were selected by Bragg reflection from an array of Heusler crystals. A pyrolytic graphite (PG) filter was placed between the sample and the analyser to suppress higher-order harmonics in the scattered beam. The neutron spin polarization **P** was maintained in a specified orientation with respect to the neutron scattering vector **Q** by an adjustable guide field of a few mT at the sample position. The data were obtained with a final neutron wavevector of 4.1 Å<sup>-1</sup>. We mounted the x = 1/3 crystal with the [001] and [110] crystal directions in the horizontal scattering plane, so that (h, h, l) positions in reciprocal space could be accessed.

Unpolarized-neutron experiments were performed on the IN8 triple-axis spectrometer at the Institut Laue-Langevin. On IN8 the energies of the incident and elastically scattered neutrons were selected by Bragg reflection from a PG (002) crystal, and a PG filter placed between the sample and the analyser suppressed the higher-order harmonics. The data were obtained with a final neutron wavevector of 2.662 Å<sup>-1</sup>. The crystal was orientated with the [100] and [010] crystal directions in the horizontal scattering plane, so that (*h*, *k*, 0) positions in reciprocal space could be accessed.

#### 4. Results

The excitation spectrum of  $La_{5/3}Sr_{1/3}NiO_4$  in the energy range 10–40 meV contains strong phonon scattering, and it is in this energy range that the 'resonance' occurs. To isolate the magnetic scattering we employed longitudinal polarization analysis with the neutron polarization **P** aligned parallel to the scattering vector **Q**. In this configuration a neutron's spin is flipped during an interaction with electronic magnetic moments, but remains unchanged when scattered by non-magnetic processes, e.g. phonon scattering [29]. The existence of the 'resonance' in the magnetic excitation spectrum was originally established by separating the spin-flip (SF) from the non-spin-flip (NSF) scattering [9, 11], as shown in figure 1(c).

In figure 2 we show the temperature evolution of the SF scattering with  $\mathbf{P} \parallel \mathbf{Q}$  at the magnetic zone centre (1/3, 1/3, 2.6) for La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub>. The magnetic scattering is proportional to the response function  $S(\mathbf{Q}, E)$  which is related by the fluctuation–dissipation theorem to the imaginary part of the generalized susceptibility [30],

$$S(\mathbf{Q}, E) = \frac{1/\pi}{\exp(-E/k_{\rm B}T) - 1} \,\chi''(\mathbf{Q}, E).$$
(1)

The data in figure 2 have been normalized by the Bose factor, i.e. the temperature-dependent factor in front of  $\chi''(\mathbf{Q}, E)$ . For bosonic excitations, like linear spin-waves, this normalization corrects for the increase in scattering that comes from the thermal population of the mode at non-zero temperatures, and allows changes in  $\chi''(\mathbf{Q}, E)$  with temperature to be identified. In reality, the corrected data are only approximately equal to  $\chi''(\mathbf{Q}, E)$  because the temperature dependence of the SF background may differ from that of the Bose factor.

The 14 K data, figure 2(a), show a prominent peak at 27 meV and are consistent with the previous measurement at this temperature, figure 1(c), which was made at a different magnetic zone centre and with a different final neutron



**Figure 2.** Temperature dependence of polarized neutron scattering from  $La_{5/3}Sr_{1/3}NiO_4$ . The measurements are energy scans with the neutron scattering vector fixed at the magnetic zone centre (1/3, 1/3, 2.6). Measurements are shown for temperatures of (a) 14 K, (b) 70 K, (c) 100 K, (d) 125 K, (e) 150 K and (f) 200 K. With the neutron polarization P parallel to the scattering vector **Q** the spin-flip data shown here are magnetic in origin. The data have been corrected for the Bose factor in the neutron cross-section.

wavevector. As the temperature is increased, the 27 meV peak is observed to shift to lower energies and the valley at around 15 meV begins to fill up.

To quantify the peak shift we have fitted the 14, 70 and 100 K scans with two Gaussian peaks on a sloping (14, 70 K) or flat background with the low-energy peak centre fixed to the value from 70 K (100 K). Based on this analysis we find that the energy of the resonance-like peak shifts from  $26.7 \pm 0.5$  meV at 14 K to  $24.9\pm2.5$  meV at 70 K and  $22.0\pm1.3$  meV at 100 K.

By 125 K the peak has almost completely filled in the valley and begun to merge with the lower-energy peak. It is difficult to extract a peak energy by systematic fitting, but from figure 2(d) it is not unreasonable to believe that the resonance peak still exists centred near 20 meV. At 150 and 200 K, figures 2(e) and (f), it is no longer possible to discern the resonance peak in the data.

To bring out the changes in the magnetic scattering with temperature we display in figure 3 the 14, 125 and 150 K data



Figure 3. The constant Q scans of figure 2 after subtraction of the 200 K data. The temperatures for each scan are (a) 14 K, (b) 125 K and (c) 150 K. In (b) the solid line represents a data fit of a Gaussian peak on a sloping background.

after subtraction of the 200 K data. At 200 K there is no static magnetic order left so this temperature is a reference for the response of the magnetically disordered system. Looking at figure 3(c) we see that at 150 K there is no change in the excitation spectrum relative to 200 K within experimental error. However, at 125 K, figure 3(b), a small positive difference signal is observed near ~25 meV. This suggests that the resonance peak develops at a temperature between 150 and 125 K. At lower temperatures the positive difference signal appears in the vicinity of 15 meV. This shows that as the resonance peak develops, intensity is transferred from energies around 15 meV to higher energies.

Having established the trends in the temperature variation of the 'resonance' we next describe the temperature evolution of the q-1D dynamic spin correlations along the charge stripes in La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub>. The scattering from these correlations is observed at relatively low energies. Previously we measured the temperature dependence of this q-1D scattering at an energy of 2 meV [25]. In low-dimensional magnetic systems the temperature dependence of the spin correlations can vary strongly with energy. Because the temperature range studied here corresponds to thermal energies much larger than 2 meV  $\approx k_{\rm B}$  (T = 20 K) we decided to re-measure the temperature dependence of the q-1D scattering at a higher energy. We chose an energy of 5 meV, at which the scattering is still relatively strong but which is larger than 2 meV by a significant factor. Constant-energy scans were made parallel to the (110) direction crossing the q-1D ridge at (0.15, 1.35, 0). At this energy and scattering vector the scattering from phonons is relatively weak and unpolarized neutrons could be used for the measurements.

Figure 4 shows the temperature dependence of the magnetic scattering from the q-1D AFM correlations at 5 meV. The data in figure 4 have not been corrected for the temperature dependence of the Bose factor in the neutron cross-section. At this energy the signal consists of two nearly resolved peaks, consistent with the dispersion measured previously [25]. In figure 4(a) the magnetic scattering intensity is observed to decrease noticeably on warming from 14 to 40 K even though  $k_{\rm B}$  (T = 40 K) < 5 meV. By 70 K the signal is about a factor 4 weaker than at 14 K. At higher temperatures, shown in figure 4(b), the intensity decreases further and the background becomes larger, especially on the right-hand side of the scan range. By 150 K there is no significant evidence of a peak in the data. The magnetic correlations from the charge stripe electrons are no longer present at and above 150 K.

To assess the change in intensity with temperature more quantitatively we fitted the 5 meV data in figure 4 with a Gaussian peak on a sloping background. The background



**Figure 4.** Temperature dependence of the quasi-one-dimensional magnetic scattering associated with the charge stripes in La<sub>5/3</sub>Sr<sub>1/3</sub>NiO<sub>4</sub>. The data were collected at an excitation energy of E = 5 meV and at a final neutron wavevector of  $k_f = 2.662$  Å<sup>-1</sup>. The temperatures are (a) 14, 40 and 70 K, and (b) 100, 125 and 150 K. The data in (a) have been scaled to the higher monitor value of the scans in (b). In the figure the data are offset by the addition of 2000 counts in (a) and 1000 counts in (b). No correction has been made for the Bose factor.



**Figure 5.** Integrated intensities of the constant energy scans of quasi-one-dimensional magnetic excitations from the charge stripes in  $La_{5/3}Sr_{1/3}NiO_4$  at E = 5 meV, where the fits have been corrected for the Bose factor.

above 100 K appears to be structured and temperaturedependent and we used a sloping background as the simplest approximation. We performed similar fits to the 2 meV data measured previously. Figure 5 shows the fitted integrated intensity variation of the scattering at E = 2 and 5 meV, corrected for the Bose factor. The results show that the temperature dependence of the q-1D magnetic response at 2 and 5 meV is quite similar. It is reasonable to assume, therefore, that correlations between spins within the stripes are almost completely destroyed by 125 K.

### 5. Discussion and conclusions

The main experimental finding of this work is that whereas the q-1D spin correlations in LSNO decrease rapidly with increasing temperature and are virtually absent at temperatures above 100 K the 'resonance' peak is still quite well defined at this temperature, albeit with a softening in its energy. This strongly suggests that the resonance is not caused by an interaction between the spin-wave-like excitations from the magnetic order and the q-1D AFM correlations along the charge stripes.

What, then, does cause the resonance peak in LSNO? There are a number of possibilities that need to be explored further.

First, the magnetic structure shown in figure 1(a) could be incorrect. If there were more than two spins per magnetic unit cell then there would be more branches to the spin-wave dispersion curve, and the resonance could be the minimum of a gapped optic mode. So far, however, there is no evidence that the magnetic structure is not that depicted in figure 1(a). Moreover, the clear existence of a resonance peak at temperatures above 100 K proves that the resonance is not the product of a magnetic transition which has been observed to occur at ~50 K [31].

Second, linear spin-wave theory might not be adequate to describe the excitations from the ground state of the stripe order due, for example, to quantum effects. Specifically, the magnetic structure can be thought of as an array of coupled S = 1 zig-zag q-1D AFM chains, and such 'Haldane' chains are expected to have a gap. The main difficulty with this idea is that the strength of the inter-chain coupling is a significant fraction of the intra-chain coupling (J'/J = 0.5) [9], which means the excitations are not expected to be particularly 1D in character. However, in some cases inter-chain coupling is actually found to increase the Haldane gap [32].

A third possibility is that the resonance arises from a coupling between spin-waves and a non-magnetic excitation. This could for example be an optic phonon of the host lattice, or a mode associated with a structural distortion around the charge stripes, or a motion of the charge stripes themselves. The first of these is unlikely because the resonance is not observed in undoped La<sub>2</sub>NiO<sub>4</sub>. In fact, the resonance is only found in La<sub>2-x</sub>Sr<sub>x</sub>NiO<sub>4</sub> in a fairly narrow range of doping around x = 1/3, the composition at which it is most prominent [11].

It is not observed at x = 1/2 [33]. On the other hand, there is already evidence that charge stripe order modifies the phonon spectrum at high energies [34, 35], so it is possible that other modes at lower energies are affected by charge order and could couple to the magnetic excitations. In this respect it may be significant that x = 1/3 is a special composition at which the charge order and magnetic order have the same periodicity (see figure 1(a)) so the magnetic resonance is at a commensurate wavevector with the charge order. The coincidence of the magnetic and charge ordering wavevectors at x = 1/3 may make it favourable for spin excitations to couple to collective motions of charge stripes at the zone centre of the charge superlattice. The existence of a commensurability gap due to pinning of the stripes to the lattice could explain why the spin excitation spectrum is most affected above 10 meV.

We will finish with a comparison of the resonance peaks in LSNO and in the cuprates as shown by the present work. We have found here that the energy of the resonance peak in LSNO decreases slightly with increasing temperature, which is similar to the behaviour of the resonance peak in YBCO whose energy also decreases slightly with increasing temperature [13, 36]. Moreover, the formation of the resonance peak in both LSNO and the cuprates is associated with a transfer of spectral weight from lower to higher energies. In the case of the superconducting cuprates, however, the transfer of spectral weight affects the excitations down to the lowest observable energy and leads to a gap in the spin excitation spectrum [4-7], whereas in LSNO the spectral weight is transferred from the region around 17 meV and the spin excitation spectrum below  $\sim 10 \text{ meV}$  does not seem to be affected by the formation of the resonance.

In summary, our measurements of the spin excitation spectrum of  $La_{5/3}Sr_{1/3}NiO_4$  indicate that the resonance-like mode in the excitations of the ordered Ni<sup>2+</sup> spins is not caused by coupling to the magnetic excitations of the charge stripe electrons. Further work will be needed to investigate other possible origins of this intriguing feature. Whether or not the resonance peaks in LSNO and the cuprates have any common underlying cause, the study of spin excitations in complex magnetic oxides with multiple order parameters continues to uncover phenomena which challenge our understanding of interacting electrons in the solid state.

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