Strain response of magnetic order in perovskite-type oxide films

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The role of elastic strain for magnetoelectric materials and devices is twofold. It can induce ferroic orders in thin films of otherwise non-ferroic materials. On the other hand, it provides the most exploited coupling mechanism in two-phase magnetoelectric materials and devices today. Complex oxide films (perovskites, spinels) are promising for both routes. The strain control of magnetic order in complex oxide films is a young research field, and few ab initio simulations are available for magnetic order in dependence on lattice parameters and lattice symmetry. Here, an experimental approach for the evaluation of how elastic strain in thin epitaxial films alters their magnetic order is introduced. The magnetic films are grown epitaxially in strain states controlled by buffer layers onto piezoelectric substrates of 0.72Pb(Mg₁/₃Nb₂/₃)O₃–0.28PbTiO₃(001). As an example, the strain dependence of the ordered magnetic moment of SrRuO₃ has been investigated. At a tensile strain level of approximately 1%, SrRuO₃ is tetragonal, and biaxial elastic strain induces a pronounced suppression of the ordered magnetic moment. As a second example, a strain-driven transition from a ferromagnetic to a magnetically disordered phase has been observed in epitaxial La₀.₈Sr₀.₂CoO₃ films.
1. Introduction

The promise of elastic strain for realizing large magnetoelectric effects in thin films is twofold, as seen today. Strained films may be both ferroelectric and magnetically ordered when the respective bulk material is not or differently ordered. In the past decade, theoretical efforts have been taken to predict ferroelectricity or multiferroicity [1–3] as a consequence of elastic strain in perovskite-type oxide films. EuTiO$_3$, a paraelectric antiferromagnet in bulk, has been found to reveal a ferroelectric and ferrimagnetic ground state in films under tensile strain [4]. A strong challenge for the discovery of strain-induced ferroic orders lies in the tendency for non-stoichiometric growth of oxide films under large strain. On the other hand, elastic strain at interfaces is the mostly used coupling mechanism in heterogeneous multiferroics to date [5–8]. The scarcity of substances with a substantial intrinsic magnetoelectric response at ambient temperatures has triggered the search for combinations of strong ferroelectric and ferro(i)magnetic substances with large coupling efficiency [5–8]. The coupling at the interfaces between magnetic and ferroelectric components works through elastic or electronic mechanisms, with record values achieved with the elastic coupling of best magnetostrictive and piezoelectric materials.

Beyond the search for strong magnetoelectricity, there is fundamental interest in strain-dependent magnetic ordering in oxides with strong electron correlation. Controlled lattice deformations may have strong impact on the balance of magnetic exchange interactions in these oxides with many coupled electronic degrees of freedom. For very few complex (i.e. non-binary) oxides, one can find theoretical simulations of the ordered magnetic moment in a strained ground state. One example is SrRuO$_3$, where the actual symmetry of the film is predicted to decide whether a complete suppression of the ferromagnetically ordered moment (strain level of about 2%) or an enhancement under tensile strain will occur [9,10]. One major obstacle is the lack of knowledge about the detailed lattice structure (the positions of atoms) of strained films, because there is no direct method available for its analysis (such as neutron diffraction for larger volumes). Oxides subject to a strong research interest such as BiFeO$_3$ have been found to undergo strain-induced structural phase transitions and reveal a strain-controlled morphotropic phase boundary [11,12]. This underlines the potential for structural transitions in strained films which may alter the magnetic order [13]. For perovskite-type oxides, a recent advance towards finding the atomic positions in strained films is based on the measurement of half-order Bragg peaks arising from collective anti-phase rotations of oxygen octahedra [14]. Comparing the measured with calculated intensities allows one to quantify the rotation angles of the octahedra in different strain states.

Based on this stage of knowledge and interest, experiments exploring the strain-dependent magnetization of transition metal oxide films are a reasonable way to identify the magnetic response to elastic strains. The mostly followed procedure is to grow films on substrates with several different in-plane lattice parameters—the static strain approach. Elastic strains of about 2\% may be reached. Higher strains may occur in the case of a structural transition, e.g. in BiFeO$_3$. A disadvantage and source of errors is the possibility of secondary strain effects. These are caused by lattice defects developing as a consequence of strain. This may be, for instance, a variation in the oxidation level. Dynamic strain has been reported using the piezoelectric or ferroelastic strains of ferroelectric substrates (BaTiO$_3$ [15,16], (1–$x$)PbMg$_{1/3}$Nb$_{2/3}$O$_3$–$x$ PbTiO$_3$ (PMN-PT) [17–19]). The electrically controlled strain from the substrate allows one to directly probe strain effects without inducing additional lattice defects in the oxides. Disadvantages are the lower magnitude of the dynamic strain and/or the non-uniformity of strain arising from the ferroelectric domains in the substrate. In this work, the combined approach of static and dynamic strain is discussed. We report on the evaluation of the strain dependences of the magnetic Curie temperature and the ordered magnetic moment in the ground state. The results and discussion part is structured as follows. Section 3a outlines the combined application of static strain from buffer layers and dynamic strain from ferroelectric substrates. The separation of different contributions to the strain response of magnetization is discussed in §3b. Sections 3c, d report on two examples: the
strain-dependent ordered magnetic moment of SrRuO$_3$ ($\S$3c) and a strain-driven magnetic phase transition in La$_{0.8}$Sr$_{0.2}$CoO$_3$ ($\S$3d).

2. Experimental

The thin films have been grown by pulsed laser deposition (KrF, 248 nm) on buffered PMN-PT(001) substrates at a growth temperature less than 750°C. Growth details for the scandate buffers have been reported previously [20]. SrRuO$_3$ has been grown at 700°C in 0.3 mbar of flowing oxygen, La$_{0.8}$Sr$_{0.2}$CoO$_3$ at 650°C in 0.2 mbar. Both types of films are cooled down in an oxygen atmosphere. Details about growth and characterization of the films will be published elsewhere. The lattice structure and the film thickness have been measured with a Philips X’Pert MRD diffractometer with CuK$_\alpha$ radiation. The magnetization has been recorded in a SQUID magnetometer (Quantum Design) using a sample holder with electrical wiring for voltage application to the substrate. The substrate voltage was supplied by a Keithley 2410 source meter with current limitation set to 1 $\mu$A. Currents of this magnitude could perturb the magnetic measurement with their magnetic field, but the typical current level during magnetic measurements is less than 100 nA. The magnetic films (whose resistivity is many orders lower than that of the substrate) served as upper electrode to the substrate, whereas the backside of the PMN-PT(001) plates covered with a gold/NiCr electrode acts as bottom electrode (figure 1a).

The PMN-PT(001) substrate with 28% PbTiO$_3$ used in our study can provide reversible linear biaxial strain of about 0.13 $\pm$ 0.01% when an electric field of 13.3 kV cm$^{-1}$ is applied perpendicular to the film plane [21], corresponding to a voltage of 400 V across the 0.3 mm thick plate. The level of strain has been recorded for a number of crystals by X-ray diffraction at 300 K. Before the strain measurements, the substrate needs to be poled carefully, increasing the voltage stepwise, at 300 K in a field of about 10 kV cm$^{-1}$. For low-temperature measurements, the sample must be field-cooled from room temperature in order to avoid slow relaxation from domain processes, because...
the low-temperature coercive field is extremely high, and no stable state of aligned domains can be reached [22]. The temperature dependence of the strain has been evaluated between 300 and 10 K [22] and is found to be weak between 250 and 50 K. At higher temperature (including 300 K), the strain gradually increases towards the Curie temperature of the substrate near 400 K. The low-temperature drop of the strain is a factor of 3–4 between 50 and 10 K, the origin of it not being well known. Care must be taken, because differences in the composition may lead to different strain levels because of the closeness of the morphotropic phase boundary of the PMN-PT.

3. Results and discussion

(a) Static and reversible strain application

The surface lattice of the strained PMN-PT(001) substrate is nearly quadratic. A refinement of the lattice structure in different fields [21] shows a monoclinic structure with a difference of 0.12% between the orthogonal $a$ and $b$ in-plane parameters. The $b$ parameter is parallel to the projection of the local polarization vector, reflecting the influence of the actual domain pattern in the substrate. For an ideally (001)-cut substrate, all four domain variants with polarizations along the space diagonal of the pseudo-cubic unit cell are equal under an applied electric field, leading to a pure polarization rotation towards the 001 direction and no movement of domain walls [23]. The strain along both the $a$ and $b$ axes is equal in the applied field range [21], and it is thus called a biaxial strain. For any material which is not significantly altered by the difference of 0.12% in $a$ and $b$, the substrate provides sufficiently uniform biaxial strain for quantitative investigation of strain dependences.

The level of the piezoelectrically induced strain is limited to several tenths of a percent in good vacuum, about 0.18% in air and 0.1% in a typical helium atmosphere of a cryostat such as that in the SQUID magnetometer because of arcing between the substrate’s electrodes. This dynamic strain level is about an order of magnitude smaller than the achievable static strains, leading to linear strain dependences in the measured strain range in most cases. However, most strain dependences are nonlinear on a larger scale of the strain. Hence, the full information on the strain dependence of magnetization can only be obtained if the dynamic strain is applied to films with different static (as grown) strain. A magnetic oxide can be grown in different strain states on PMN-PT or other substrates through utilization of compositionally tuned buffer layers. Two available buffer systems for magnetic films are LaAl$_{1-x}$Sc$_x$O$_3$ (LASO) [20] and Ba$_{1-x}$Sr$_x$TiO$_3$ [24]. The composition $x$ allows one to approximately predefine the desired lattice parameter and prepare a film series in selected strain states. The function of the buffer requires that it is grown in relaxed state onto the substrate, whereas the magnetic oxide to be studied needs to be grown coherently onto the buffer. Figure 1b shows as an example the reciprocal space map of a La$_{0.8}$Sr$_{0.2}$CoO$_3$ film grown nearly coherently on a tuned LASO buffer on PMN-PT(001). The additional SrTiO$_3$ layer under the buffer layer is required to prevent interdiffusion, because we found that LaAlO$_3$ cannot be grown well on PMN-PT. We note that the number of layers does not pose a problem to the strain transfer from the substrate into the magnetic film, unless the structural quality of the films is compromised. Films with cracks, voids or pronounced columnar growth mode may lose the epitaxial elastic coupling at interfaces. Smooth continuous films will be fully strained unless plastic defect formation occurs. The latter is not likely in perovskite-type oxides at temperatures $T \leq 300$ K because of the high energies for defect formation. The ultimate proof of strain transfer from substrate to film can be obtained by X-ray diffraction. We found that superlattices of La$_{0.7}$Sr$_{0.3}$MnO$_3$/SrTiO$_3$ with 100 interfaces and thicknesses up to 500 nm show the full strain level like the substrate [19].

(b) Contributions to the strain response of magnetization

The direct strain dependence of the magnetization can be measured as $M(\varepsilon(E))$ with the piezoelectrically induced biaxial in-plane strain $\varepsilon$ by changing the electric substrate field $E$. 
Because the relative change of $M$ is small due to the small applied strain, the evaluation of the result is more clear using the relative change $\Delta M / M = [M(E) - M(E = 0)]/M(E = 0)$. For example, figure 2a shows $\Delta M / M$ measured on a La$_{0.7}$Sr$_{0.3}$CoO$_3$ film for a fixed applied field at various temperatures. Values of a few per cent are typical and reflect a substantial strain response, because the corresponding strain level is of the order of 0.1%. The data in figure 2a are recorded in a constant magnetic field of 100 mT that is sufficient to saturate the magnetization in the ferromagnetic film above $T = 50$ K. We note that bulk La$_{0.7}$Sr$_{0.3}$CoO$_3$ is a cluster glass [25] with imperfect ferromagnetic order. The small amount of magnetic disorder prevailing in the ‘ferromagnetic phase’ is not seen in films, probably owing to the small relative volume. Hence, $\Delta M / M$ reflects the real change of the local magnetization value, because no domains exist and might be altered by the strain application. This is a beneficial condition that cannot always be achieved for a measurement of strain-dependent magnetization. The magnetic saturation field may be too high or the existence of multiple structural domains may prevent the identification of a global magnetic easy axis. Another problem may be posed by field-induced magnetic order, so that measurements should be taken in remanence. In the case of multiple magnetic domains in the film, one needs to evaluate the effect of strain on the domain configuration for a meaningful interpretation of $\Delta M / M$ data. The measured $M$ is an average over all domains and reflects

Figure 2. (a) Strain-induced relative change of the magnetization, $\Delta M / M$, of a La$_{0.7}$Sr$_{0.3}$CoO$_3$ film measured along an in-plane easy axis. The arrow marks the independently measured Curie temperature. (b) Strain-induced relative change of the magnetization, $\Delta M / M$, of a SrRuO$_3$ film under 1% of tensile strain measured along an in-plane easy axis. (Online version in colour.)
strain-induced shifts of domain walls as well as strain-driven magnetization rotations. These contributions must be circumvented if one looks for the strain-dependent value of the ordered magnetic moment. In order to avoid strain-driven domain modifications, the magnetic anisotropy of the film needs to be known. Fortunately, many oxides show dominating biaxial in-plane anisotropy with two orthogonal in-plane easy axes. Then, easy axes are not changed by applying biaxial strain. In this case, a measurement of the strain-dependent remanent magnetization along an easy axis will not be influenced by domain changes and gives a correct value of $\Delta M/M$ representing the real change of the ordered magnetic moment.

The spontaneous magnetization $M_S$ (being proportional to the ordered magnetic moment) of a ferromagnet depends on temperature as

$$M_S(T, a) = M_S(T = 0, a)f\left(\frac{T}{T_C(a)}\right),$$

(3.1)

with the Curie temperature $T_C$ and the in-plane lattice parameter $a$. The function $f$ varies slightly among materials [26]. The spontaneous magnetization in the ground state, $M_S(T = 0, a)$, and $T_C$ may depend on strain. Both dependences are of strong fundamental interest. The function $f$ may be strain-dependent, too, but unless some strain-driven transition occurs we assume that this effect can be neglected for the small level of the dynamic strain. The strain effect on $M_S(T = 0, a)$ can be seen directly at any temperature $T \ll T_C$, because the function $f$ converges to the value 1 there. This strain effect on the ordered ground state moment provides a constant contribution to $\Delta M/M$ (figure 3b).

**Figure 3.** (a) Schematic graph presenting two magnetization curves in two strain states with slightly shifted Curie temperature and the resulting relative difference $\Delta M/M$. (b) Schematic graph presenting two magnetization curves in two strain states with changed value of $M_S(T = 0)$ (as a factor) and the resulting relative difference $\Delta M/M$. (Online version in colour.)
The strain-driven change of $T_C$ results in a peak of $\Delta M/M$ near $T_C$ as illustrated in figure 3a. One can show mathematically that the following procedure gives a correct estimation of $\Delta M/M$ close to $T_C$ if the strain has shifted $T_C$ by $\Delta T_C$: shift the temperature of the magnetization data $M(T)$ measured under the same conditions by an assumed $\Delta T_C$ and subtract these from the original data to obtain $\Delta M(T)$. $\Delta T_C$ is adjusted, so that the measured strain-dependent data $\Delta M/M(T)$ in a range near $T_C$ agree with this calculation. In this way, $dT_C/da$ at the present inplane parameter $a$ of the film can be estimated. Figure 2a shows with La$_{0.7}$Sr$_{0.3}$CoO$_3$ a magnetic oxide that does not change $M_S(T = 0)$ under strain. $\Delta M/M$ vanishes at low temperatures and peaks near $T_C$. (We note that errors get large very close to $T_C$ for the reduced magnitude of $M$. Therefore, the data are restricted to the shown temperature range of $T < T_C$.) The example of strained tetragonal SrRuO$_3$ in figure 2b shows both contributions from a changed ground state moment and a shifted $T_C$. It will be discussed in more detail below. Both strain effects add up to the observed $\Delta M/M$.

(c) Strain-dependent magnetic moment of SrRuO$_3$

SrRuO$_3$ is a metallic ferromagnet with $T_C \sim 160$ K and an ordered magnetic moment of approximately 1.1 $\mu_B$ per formula unit. The structure at $T \leq 300$ K is orthorhombic GdFeO$_3$ perovskite-type with the lattice parameters $a_O = 5.567$ Å, $b_O = 5.5304$ Å and $c_O = 7.8446$ Å. The rotation pattern of the oxygen octahedra is described by the Glazer notation $a^-a^+c^-$. A calculation using density functional theory [9,10] predicts a strong impact of biaxial epitaxial strain on the ordered magnetic moment. In the original orthorhombic symmetry, tensile strain enhances $M_S$ to approximately 1.4 $\mu_B$ per formula unit, whereas in tetragonal symmetry, a complete suppression of magnetic order ($M_S = 0$) occurs for 2% strain of either sign. (We note that tetragonal symmetry in that calculation implies zero rotations of the oxygen octahedra. This is not necessarily fulfilled when a grown film shows tetragonal lattice symmetry.) The reason for this can be understood as a Ru low spin state induced by strong distortion of the oxygen octahedra [9]. Hence, an experiment probing the real strain-dependent magnetic moment of SrRuO$_3$ seems interesting.

Epitaxial films of SrRuO$_3$ (40nm) have been grown in predefined strain states on PMN-PT(001) and SrTiO$_3$(001) substrates using Ba$_{1-x}$Sr$_x$TiO$_3$ buffers. Films under 1% tensile strain have a strain-induced tetragonal structure with the $c$-axis along the film normal (figure 4a) as found by X-ray diffraction and angular-dependent magnetoresistance measurements. Hence, the film in the tensile strain state is structurally single-domain. The magnetic anisotropy changes completely between a compressively strained film on SrTiO$_3$(001) showing a tilted out-of-plane easy axis [27] and tetragonal films showing biaxial in-plane anisotropy with $\{110\}_t = \{100\}_p$ easy axes (figure 4c). (Subscripts ‘t’ and ‘pc’ refer to tetragonal and pseudo-cubic indices, respectively.) The substantial difference of the magnetizations measured in-plane and out-of-plane at the maximum field of 4.5 T (figure 4b) reveals the lack of saturation and the likely presence of domains. However, the biaxial in-plane anisotropy allows one to measure the correct strain dependence $\Delta M/M$ as discussed in §3b. Figure 2b presents the data recorded in remanence after cooling in an in-plane magnetic field $\mu_0H = 0.1$ T/$100_p$. The total $\Delta M/M$ has been fitted with a contribution from a $T_C$ shift and a constant contribution from a change of $M_S(T = 0)$. We obtain $dT_C/da = -6.2$ K$^{-1}$ for $a = 3.96$ Å of this film, in contrast to the known enhancement of $T_C$ with tensile strain at $a = 3.93$ Å in a previous work [24]. This indicates the presence of a maximum of $T_C(a)$ between the investigated values of $a$. The ordered moment decreases under tensile strain such as $1/M_S dM_S/da = -11%/1%$. This relative change agrees well with the calculation for the tetragonal structure in [9]. We conclude that, at a tensile strain level of approximately 1%, SrRuO$_3$ is tetragonal, and biaxial elastic strain seems to distort the oxygen octahedra, leading to an efficient suppression of the ordered magnetic moment.

(d) Elastically driven phase transition in La$_{0.8}$Sr$_{0.2}$CoO$_3$

La$_{1-x}$Sr$_x$CoO$_3$ is ferromagnetic with a Sr doping level of $x \geq 0.18$, whereas the undoped LaCoO$_3$ is non-magnetic with a Co$^{3+}$ low spin state [25,28,29]. Sr induces magnetic clusters with higher
Figure 4. (a) Orientation of the unit cell of tetragonal SrRuO$_3$ on the (Ba,Sr)TiO$_3$ buffer layer. (b) Magnetization loops taken at 10 K in out-of-plane direction (001)$_{pc}$ and in an easy axis direction in the plane, (100)$_{pc}$. (c) Angular-dependent magnetization in the film plane measured in a magnetic field of 1.5 T. The four maxima indicate the easy axes along (100)$_{pc}$. (Online version in colour.)

spin states [29] that grow in number and size with increasing $x$, thereby forming a spin glass phase below the phase boundary of $x = 0.18$ where percolation and long-range ordering of the ferromagnetic phase occur [25]. We selected the composition of $x = 0.2$ close to the phase boundary in an attempt to drive the transition by elastic strain.

Thin films of 40 nm thickness have been grown on PMN-PT(001) in a wide range of in-plane strain, using the LASO buffer system ($§3a$). Their lattice symmetry is tetragonal owing to the epitaxial constraint, in contrast to the rhombohedral bulk lattice. Figure 5 demonstrates the linear elastic response of the films with decreasing tetragonality $t = c/a$ from 1.25 to 0.97 and associated increasing volume of the unit cell. The estimated Poisson number is $\nu = 0.31$. We note that it is not clear at this point whether the films keep the same composition, such as the oxygen content, in all strain states, because La$_{1-x}$Sr$_x$CoO$_3$ is known for strain-dependent oxygen deficiency [30].
Figure 5. Tetragonality and volume of the unit cell calculated from the tetragonal lattice parameters $a$ and $c$ of strained La$_{0.8}$Sr$_{0.2}$CoO$_3$ films. Samples are named alphabetically in the sequence of their in-plane lattice parameter. (Online version in colour.)

The magnetic anisotropy of all films is found to be biaxial in-plane with $\{110\}_{\text{pc}}$ easy axes. The saturated magnetization ($M_{\text{sat}}$) at 10 K in a magnetic field of 4.5 T reveals a strong reduction of ordered magnetic moment with growing in-plane lattice parameter (figure 6a, triangles). We note that this reduction is unlikely to result from a lowering of the Co spin state, because tensile strain is known to enhance the magnetic moment in LaCoO$_3$ [31]. Hence, the reduction of $M_{\text{sat}}$ indicates a growing magnetic disorder. This is also reflected by a decrease of $T_C(a)$ (figure 6a) with increasing tensile strain. Three ranges of the lattice parameter are distinguished in figure 6a: region I with compressive strain, region II with moderate tensile strain and gradual drop of $T_C$ and region III of larger tensile strain with strong drop of $T_C$ and scattering among samples. Taking the reduction of $M_{\text{sat}}$ and the known doping-dependent phase diagram (reproduced in figure 6b) into account, we suggest that the percolation limit of the ferromagnetic phase is reached at the boundary between regions II and III, and region III contains spin glass samples. The scattering of $T_C$ would naturally result from differences in the microstructure of the samples, because samples A–G and H–I have been grown in different runs.

The dynamic strain is used to validate the strain-driven nature of the observed induced disorder with increasing lattice parameter. The heterogeneous magnetic state of the samples (comprising ferromagnetic and spin glass phase) makes the interpretation of measured $\Delta M/M$ data more difficult. Fortunately, the spin glass contribution can be ignored in a temperature range of $T_{\text{FM}}^C > T > T_{\text{SG}}^C$ (with the ordering temperatures $T_{\text{FM}}^C$ and $T_{\text{SG}}^C$ of the ferromagnetic and the spin glass phase, respectively). This allows us to derive $dT_C/da$ values for the region II (as described in §3b), whereas, in region III, no meaningful values of $dT_C/da$ can be obtained, because $T_{\text{FM}}^C$ is similar to $T_{\text{SG}}^C$. The slopes $dT_C/da$ are inserted in figure 6a and agree well with the $T_C(a)$ dependence in region II. We note that sample B in region III is compressed; hence, the opposite slope there indicates suppression of $T_C$ by compressive strain. The comparison of the static and the dynamic strain response of $T_C$ (in region II) allows one to clarify the strain-driven nature of the magnetic disordering. Even though this argument cannot be extended rigorously to region III, the unchanged elastic response of the lattice structure up to at least the sample H (figure 5) is in favour of an unchanged composition also in this region of strain. We conclude that an essentially strain-driven transition from a ferromagnetic to a magnetically disordered phase has been observed in La$_{0.8}$Sr$_{0.2}$CoO$_3$ films.

4. Summary

An experimental approach for the evaluation of how elastic strain in thin epitaxial films alters their magnetic order has been introduced. The role of elastic strain for magnetoelectric materials
and devices is twofold, because strain can induce ferroic ordering in thin films and can be used for coupling a magnetic and a ferroelectric phase. Our experimental approach could be useful for testing the strain response of magnetic substances. It can provide deeper insights into the relationship of structure and magnetic order in transition metal oxides with strong electron correlation which is still difficult to access with fundamental theory. As an example, the strain dependence of the ordered magnetic moment of SrRuO₃ has been investigated. SrRuO₃ is found to undergo a structural transition to a tetragonal phase at medium tensile strain associated with a strain-induced drop of the ordered moment indicating distortion of the oxygen octahedra in the perovskite-type lattice. As a second example, the strain-driven suppression of the ferromagnetic phase in La₀.₈Sr₀.₂CoO₃ has been addressed.

References


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