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Au-induced perpendicular magnetization in Fe films grown on Si(001)

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Abstract
We report on Fe films grown on Au-buffered Si(001) showing perpendicular magnetization at ambient temperatures. The in-plane magnetization in ultra-thin Fe films grown at 150 K reorients irreversibly out-of-plane as a result of annealing close to room temperature. A Au cap layer which is expected to segregate to the top of the Fe film may be responsible for this behaviour. This hypothesis is based on the observation of an in-plane to out-of-plane reorientation of magnetization upon the growth of an ultra-thin Au cap layer on a slightly thicker Fe film. The origin of such a strong out-of-plane anisotropy of Fe induced by Au is discussed.

1. Introduction

The possibility of realizing devices by exploiting the electron's property of possessing a spin has captured the attention of the scientific community since the past decade [1–4]. There are basically two aspects which make the issue of perpendicular magnetization a practical requirement, although not a prerequisite for spintronic devices. First, high-density information storage media can be obtained, and second, circularly polarized photons can be emitted perpendicular to the surface plane as the result of spin-injection and electron–hole recombination in a direct band-gap semiconductor.

However, the metal–semiconductor interface is generally not very sharp after growth or storage at ambient temperatures [5–8], as the dangling bonds on semiconductor surfaces increase its reactivity. The resulting preferential bonding of a ferromagnetic metal adatom to the substrate's dangling bonds seems to favour an in-plane magnetic anisotropy over a wide temperature range [8, 9], or even to completely kill the magnetization in the first monolayers (MLs) (see for instance [10], [11]). Therefore, to achieve a state with perpendicular orientation of magnetization, one needs either to reduce the growth [10] and operating temperature, or to employ a passivating buffer layer [11]. Nonetheless, the former case is not a practical one, since commercial devices need to be operated at ambient temperatures. Artificially fabricated alloys exhibiting perpendicular magnetization [12] at ambient temperature may offer an alternative solution.

We recently showed that the out-of-plane ferromagnetic order in ultra-thin Fe films on Au-covered Si(001) was observed within about 1 ML of Fe above the magnetization onset coverage (≈1.5 ML at 150 K and ≈4 ML at 300 K) [11]. By Au-covered Si(001), we designate the Au-induced reconstructed Si(001) [13–16], subsequently covered with ≈1.5 ML of Au at ≈150 K. In this way, we expected that the native Si(001) reactivity may be reduced, by partly satisfying the surface dangling bonds. Concerning the thermal stability, we proved that a 2.3 ML thick Fe film preserves the perpendicular orientation of magnetization upon annealing close to room temperature (RT) [11]. If the Fe thickness of the ≈150 K grown film approaches 3 ML, the magnetization reorients from the perpendicular to the in-plane direction [11]. This means that, for this particular thickness, the reorientation temperature equals the growth temperature [17]. This behaviour is due to a reduced influence of the surface

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anisotropy in comparison to the one of the dipole interaction as the film thickness is increased.

The observed perpendicular magnetization can be accounted for, in principle, by pondering the competing anisotropy terms: while the surface anisotropy favours a perpendicular orientation of magnetization, the shape anisotropy tends to rotate the magnetization into the film plane [18]. The bilayer structure of the Fe/Au system demands however, the consideration of structural/compositional contributions, too.

The aim of this paper is to give more insights into the phenomena enabling the manipulation of magnetization direction in an Fe/Au bilayer grown on Si(001). The ultra-thin Au film deposited on Si(001) prior to Fe film growth is able to produce the desired perpendicular orientation of magnetization at ambient temperatures with no magnetically dead layers [11]. The effect of annealing in ≈3 ML thick Fe films grown at ≈150 K and the influence of a Au cap layer is investigated.

2. Experiment

The sample investigation was done with respect to their chemical composition by Auger electron spectroscopy (AES), structure by low energy electron diffraction (LEED), morphology by scanning tunnelling microscopy (STM), and magnetic behaviour by magneto-optic Kerr effect (MOKE). In the MOKE set-up used in this work, a unipolar magnet piece is brought close to the sample, whose axis makes a non-zero angle with respect to the sample's surface. Both in-plane and out-of-plane MOKE signal can be detected. Since for out-of-plane orientation of magnetization the ellipticity is by an order of magnitude larger than for in-plane loops, large MOKE signal indicates an out-of-plane orientation of magnetization. The maximum DC magnetic field reachable with this set-up does not exceed 30 mT. Sample preparation and characterization was carried out *in situ* under ultra high vacuum (UHV) conditions at a base pressure better than 8×10^{-11} mbar. Small pieces cut from p-type Si(001) wafers have been used as substrates. Prior to their mounting into Ni contaminant free holders and inserting into UHV, the crystals were ultrasonically cleaned and then boiled for several minutes in absolute pure ethanol. After transferring them into the UHV chambers, the Si(001) substrates were first outgassed and then flashed up to ≈1600 K to remove the native oxide layer. The substrates were considered clean only after the dimer rows of the reconstructed surface were clearly seen in STM scans. The absence of any traces of carbon and oxygen in the AES spectra along with a sharp two domain (2×1) LEED pattern of low background indicated also a clean sample. The Fe (99.99% purity) deposition was performed by means of an electron beam evaporator at a growth rate of ≈1.5 ML min^{-1}, where the ML coverage is defined in terms of the atomic density of bcc Fe(001), i.e. 1.22×10^{15} atoms cm^{-2}. Au was evaporated by electron bombardment from a Au filled Mo crucible with growth rates of ≈0.5 ML min^{-1} (1 ML Au = 1.20×10^{15} atoms cm^{-2}). The low temperature (LT) growth was performed by cooling the sample holder with liquid nitrogen.

3. Results

3.1. Substrate preparation

The Si(001) surface strongly reacts with the deposited Fe resulting in an amorphous and poorly defined interface associated with the formation of silicides even at ambient growth temperatures [7]. To reduce this reaction and enable us the manipulation of the magnetization direction, we performed a passivation of the Si(001) surface by depositing slightly less than 1 ML of Au at temperatures around 1000 K. As reported earlier [14, 15], this results in a drastic modification of the Si(001) surface. The rearrangement of the surface atoms in stripes running along the ⟨110⟩ directions gives rise to the occurrence of different structures, according to the substrate temperature and the amount of deposited Au [13–16].

Shimakura *et al* [15], deduced from STM data that the c(18×2) phase occurs at ≈1000 K. A mixture of (5×3) and $(\sqrt{26} \times 3)$ ($\sqrt{26} = 5.099$) surface structures (3.84 Å unit mesh) built from both Si and Au atoms was shown by Lin *et al* [14] to form on the surface around 1100 K deposition temperature. Recently, the incommensurate (5×3.2) phase was also shown to occur under similar conditions on a vicinal Si(001) surface [16].

The two domain (2×1) LEED pattern of clean Si(001) transformed upon Au deposition at temperatures of interest here (1000–1100 K) into different ones (figures 1(*a*) and (*b*)), due to new phase formation associated with surface restructuring. The overall symmetry deduced from the LEED patterns appears quite sensitive to the growth conditions. If temperatures around 1000 K are employed to perform the reconstruction, a typical two domain $(n \times 2)$ pattern was observed here (figure 1(*a*)), while an overall (5×3) structure results if the deposition is performed at 1100 K (figure 1(*b*)). Since sharp spots can be distinguished in addition to the integer ones, it appears that the surface phases arrange themselves in a rather ordered manner to build up the reconstructed surface.

The high resolution STM images shown in figures 1(*c*) and (*d*) correspond to the deposition conditions in figures 1(*a*) and (*b*), respectively. A small difference in the deposition temperature leads to different surface structures and morphologies as the result of different surface diffusion lengths [15]. The periodicity arising from the stripe decoration and their lateral separation gives rise to the complex LEED patterns shown in figures 1(*a*) and (*b*).

In the case of 1100 K deposition temperature, from the sequences of additional spots at one and two third order positions one can judge on the coexistence of several phases. We note that the stripes in the STM image shown in figure 1(*d*) appear lying next to each other or separated either by trenches or by differently decorated stripes. Such sequences of stripes and trenches were not reported by Lin *et al* [14] in their work. It is reasonable to assert that not only the (5×3) and $(\sqrt{26} \times 3)$ phases contribute to the surface reconstruction observed here.

3.2. Growth and magnetic properties of ultra-thin Fe films

The perpendicular manipulation of magnetization direction is achieved by the growth of an additional 1.5 ML thick Au buffer layer on the Au-induced reconstructed surface of Si(001) at LT. As we reported earlier [11], this layer, and the subsequent Fe

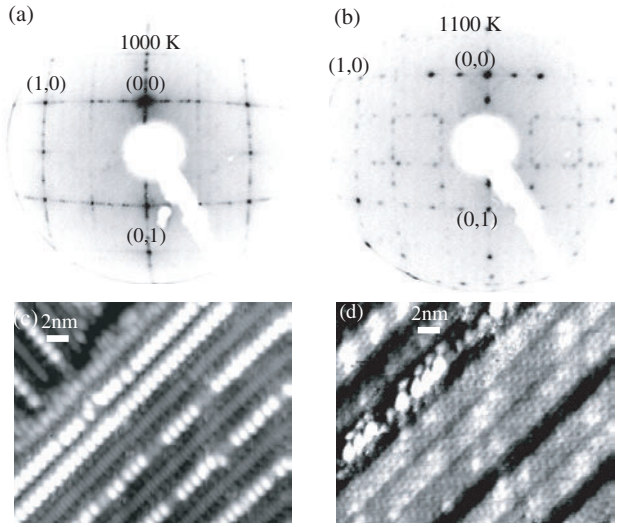

Figure 1. LEED patterns and 25 × 20 nm^2 STM images of the reconstructed surface performed by the deposition of ≈1 ML of Au on Si(001) at 1000 K (*a*) and (*c*), and 1100 K (*b*) and (*d*). The LEED images in (*a*) and (*b*) were taken at beam energies of 95 eV and 78 eV, respectively. The STM scans were performed at 45° with respect to ⟨110⟩ (the direction of stripes), at −1.6 V and 1.0 nA in (*c*) and −1.6 V and 0.6 nA in (*d*).

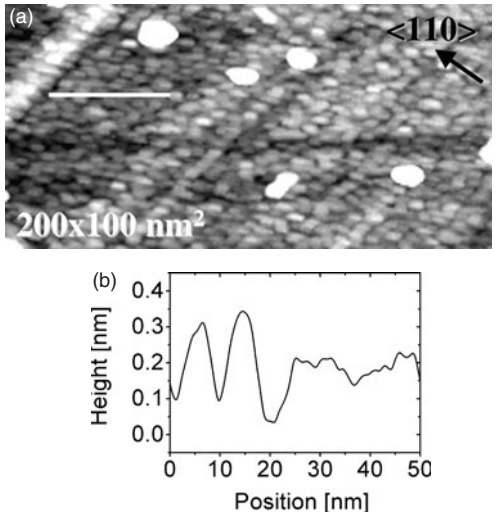

Figure 2. (*a*) 200 × 100 nm^2 STM image of ≈2.5 ML of Fe grown at LT on Au-covered Si(001). A line profile taken along the white line in (*a*) is shown in (*b*). The scan was performed at 1.6 V bias voltage and a constant tunnelling current of 1.0 nA.

film do not show a long range crystallographic order, as we deduced from the lack of any LEED pattern. Moreover, our experimental results (figure 2(*a*)) reveal the granular character of the film, but there is no indication of any texture. Earlier, it was demonstrated that a monoatomic Au cap layer is expected to segregate on the top of the film due the lower surface free energy of Au with respect to that of Fe [19].

The stripes belonging to the underlying Au-induced reconstructed surface are still visible after about 2.5 ML of Fe were grown at LT on Au-covered Si(001) (figure 2(*a*)). Small coalesced islands of 1–2 ML height (figure 2(*b*)) were observed in the STM images, but some Au crystallites can be seen as well (the white spots).

We recently found that the Fe films grown on these substrates are perpendicularly magnetized at thicknesses between 1.5 and 2.5 ML at 150 K, and in a narrow range around 4 ML for RT grown films [11]. This is in contrast to the finding of Bader and his coworkers [19, 20], who observed the perpendicular orientation of magnetization in Fe films grown on Au(001) only at LTs. The LT grown Fe/Au/Si(001) system was investigated with respect to its magnetic behaviour upon annealing close to RT. Lately, we have shown that the perpendicular orientation of magnetization in 2.3 ML Fe/Au/Si(001) is preserved upon annealing to 280 K [11].

In the following, we focus on slightly thicker films, grown at LT. An as-grown 3 ML thick film is in-plane magnetized, as we deduced from the measurement of a low-ellipticity hysteresis (loop 1 in figure 3(*a*)) The high-ellipticity hysteresis (loop 2 in figure 3(*a*)) measured upon raising the temperature to about 270 K indicates that the film undergoes a transition to a state with out-of-plane magnetization. Since a transition from the in-plane to the out-of-plane magnetization in a film which does not suffer any structural and/or chemical transformation upon raising the temperature is thermodynamically prohibited [18], such a behaviour suggests that modifications involving the Au buffer layer have to be regarded. An even stronger out-of-plane Kerr ellipticity was measured as temperature was decreased back to ≈150 K (loop 3 in figure 3(*a*)), and, with slightly increasing the thickness, magnetization flipped back into the film plane.

Let us now consider the case of a ≈3.2 ML thick Fe film grown at LT, whose direction of magnetization lies in the film plane. The corresponding low-ellipticity square-like loop is represented with a solid line in figure 3(*b*) and marked as 1. Upon annealing up to 280 K, the film appears to have preserved the in-plane orientation of magnetization as deduced from the measurement of a low-ellipticity hysteresis (loop 2 in figure 3(*b*)). The loop is strongly elongated and saturates by applying fields above 10 mT at more than 100 μrad. Therefore, it seems that magnetization was rotated out-of-plane by increasing the applied field, slightly misoriented

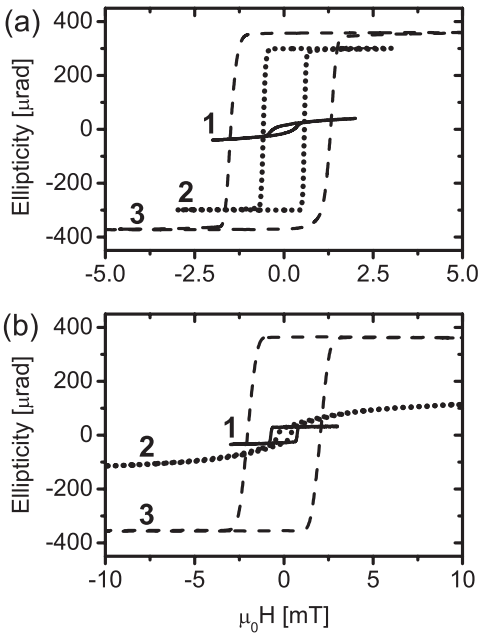

Figure 3. (*a*) An in-plane magnetized 3 ML thick Fe film grown on Au-covered Si(001) at 150 K (loop 1), undergoes a transition to a state with perpendicular magnetization at 270 K (loop 2), which is preserved upon cooling back to 150 K, where a larger signal is measured (loop 3). (*b*) The in-plane direction of magnetization in a ≈3.2 ML thick Fe film grown on Au-covered Si(001) at 150 K (loop 1) is preserved upon annealing at 280 K (loop 2) and reorients out of the film plane after ≈1.5 ML of Au is deposited at the same temperature (loop 3).

with respect to the film plane. This result is in contrast to the observation of a clear out-of-plane MOKE signal in the case of a slightly thinner Fe film annealed at ≈270 K, as described in the previous paragraph. The film was afterwards capped with about 1.5 ML of Au at 280 K, and the out-of-plane orientation of magnetization was retrieved, as we deduced from the measurement of a high-ellipticity hysteresis (loop 3 in figure 3(*b*)).

4. Discussion

To acquire valuable insights into the mechanisms leading to perpendicular magnetization, the behaviour of in-plane magnetized films grown at LT was investigated here with respect to annealing close to RT. The evolution of magnetization direction with both annealing temperature and Fe coverage for LT-grown films is sketched in figure 4. The diagram refers to four distinct cases, labelled from *A* to *D*, corresponding to four different films of increasing Fe thickness. In all these four situations, the growth conditions were kept unchanged. The case of a 2.3 ML thick film (labelled *A* in figure 4) is discussed in [11], and depicts the preservation of the perpendicular orientation of magnetization upon annealing. Case *D* corresponds to the trivial situation of a rather thick film (above 4 ML), whose in-plane direction of magnetization is kept regardless of further processing. Cases *B* and *C* picture two distinct situations, in which the direction of magnetization is altered as a consequence of thermal annealing and capping. They are discussed in the following.

An in-plane magnetized 3 ML thick Fe film grown at LT on Au-covered Si(001), (loop 1 in figure 3(*a*)), was found to undergo a transition to a perpendicular magnetization state as the temperature increased up to ≈270 K (loop 2 figure 3(*a*), and case *B* in figure 4). If this transition was not accompanied by structural and/or compositional changes, it would have been thermodynamically forbidden. The reason is that the state with perpendicular magnetization has a lower entropy than the state with in-plane magnetization [18]. Taking into account that our film shows no long range crystallographic order before and after annealing, the most likely temperature-induced processes that are able to influence the interface anisotropy are related to short range phenomena like atomic exchange, and intermixing at the Au/Fe interface. But this would lead to the interface degradation and, quite oppositely, the out-of-plane interface anisotropy is destroyed, as suggested by Liu and Bader [21]. However, it was recently proven that during the growth of Fe on Au, a ML of Au segregates to the Fe film surface [22, 23]. Accordingly, one can infer that the out-of-plane magnetization which takes over after the film was annealed close to RT might be related to the presence of Au on the top. The Au floating mechanism was shown to be dictated by the growth mode of Fe on Au [22, 23]. Nevertheless, the diffusion is enhanced as the temperature is increased. The presence of defects like island boundaries in our granular films (see figure 2(*a*)) hints at the possibility of Au-segregation [24]. We cannot rule out that also in the case of thinner films (e.g. 2.3 ML of Fe), some floating Au does not play a role in establishing and stabilizing the perpendicular magnetization.

The above hypothesis is supported by following the magnetization behaviour in a slightly thicker (3.2 ML) Fe film, grown at ≈150 K on Au-covered Si(001). The film is initially in-plane magnetized (loop 1 in figure 3(*b*)), and annealing close to RT is not able to induce a clear perpendicular magnetization (loop 2 in figure 3(*b*), and case *C* in figure 4). Under the hypothesis of Au segregation-induced perpendicular magnetization, this result infers that the segregation mechanism is hindered in the case of the 3.2 ML thick Fe film, because the pinhole-like microscopical defects might be partly closed. It is known that the amount of gold floating on the surface decreases with increasing the Fe film thickness [23]. To increase the amount of Au at the top of

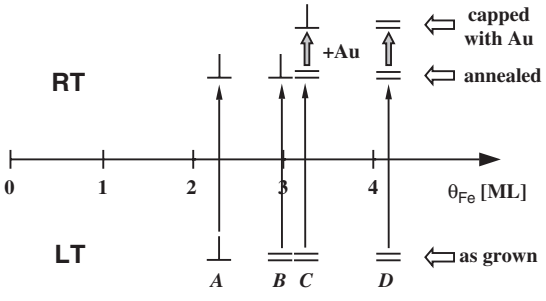

Figure 4. Diagram of magnetization direction in LT as-grown, annealed, and Au-capped ultra-thin Fe films for different film thicknesses. Symbols '⊥', and '=' stand for perpendicular and in-plane magnetization, respectively. The bottom half of the diagram corresponds to the measurement temperatures of about 150 K, while the top one to temperatures close to RT. The four distinct cases are labelled *A* (taken from [11]), *B*, *C*, and *D*, respectively. Films capped with Au are marked as '+Au'.

the Fe film, which might be required to rotate the direction of magnetization perpendicular to the film plane, we deposited a little amount of Au, and the desired effect is obtained (loop 3 in figure 3(*b*), and case *C* in figure 4). This is an evidence that the perpendicular orientation of magnetization (in the Fe film thickness range around 3 ML) is promoted, if not induced, by a Au layer present on the top of the Fe film. Our findings remain in agreement with the earlier observations of Chappert *et al* [25], who reported on the perpendicular anisotropy in a 2.5 ML Fe film grown on Au(111), induced by covering with less than 1 ML of Au.

The simplest interpretation of the perpendicular anisotropy induced by the Au cap layer, relates to an enhancement of the surface anisotropy (the UHV/Fe interface is replaced with the Au/Fe one), which is able to overcome the shape anisotropy in a rather thick disordered Fe-film. In contrast, Heinrich *et al* ([26] and references herein) found a lower surface anisotropy for the Fe(001)/Au interface (0.40–$0.54 \times 10^{-3}\,\mathrm{J\,m^{-2}}$) than for the Fe(001)/vacuum one ($0.96 \times 10^{-3}\,\mathrm{J\,m^{-2}}$) at RT, but of the same sign, which points at the normal direction to the film plane as the easy direction of magnetization. A slightly higher value was found by Elmers and Gradmann [27] in the case of Fe(110)/Au ($0.72 \times 10^{-3}\,\mathrm{J\,m^{-2}}$), but still smaller than the Fe-vacuum surface anisotropy. Therefore, one expects that the perpendicular orientation of magnetization is less favourable in the case of Fe–Au interface in comparison to the Fe-vacuum one, which does not stand for the systems investigated here. For granular films with no long range crystallographic order on buffered Si, the Au–Fe interface anisotropy seems to have a stronger interface anisotropy than the Fe–vacuum interface. A possible reason can be that the disordered films are more open or less densely packed.

The special role Au plays in the onset of the perpendicular magnetic anisotropy in ultra-thin Fe films is unquestionable, both from structural and electronic point of view [12, 28–30]. The investigation performed by Hernan *et al* [31] demonstrated that intermixing occurs at RT. More precisely, a mechanism based on the place exchange between the Fe and Au atoms and Au diffusion was proposed to explain the growth of Fe on Au(001). In addition, a recent *ab initio* study of the energetics of surface alloying on the atomic scale [32] demonstrated that site exchange between 3d transition metal impurities on Au(001) and Au atoms is energetically favourable.

Therefore, it is reasonable to suggest that the origin of the perpendicular magnetization we observed resides on the local atomic arrangement in the open and disordered Fe film, which in turn is established by the place exchange mechanism between the Fe and Au atoms [31]. This mechanism can lead to an important reorganization of the Fe layer. For instance, Fe monoatomic platelets, which have exchanged positions with the Au underneath, can be formed. The immediate result of this atomic exchange is an increased number of Fe–Au 3d–5d hybrid bonds [29] directed along the surface normal. This might lead to an enhancement of the perpendicular magnetic anisotropy, like in the case of Co/Pt(111) [33]. The positive contribution of the band energy found by Szunyogh *et al* [28] indicates that the spin–orbit coupling favours the perpendicular orientation of magnetization. The leading contribution to the anisotropy was shown to arise from the Fe layer placed just at the Fe/Au interface, pointing out the important role of the Au substrate for the actual magnitude of the surface magnetic anisotropies [28]. The suggested origin of the Au effect on the perpendicular anisotropy of Fe points at the spin–orbit coupling in Au, which forces the spins of the d electrons in the Fe layer to be more confined toward the surface normal.

5. Summary and conclusions

In this paper, we present a way to engineer the direction of magnetization in ultra-thin films grown on the technologically relevant (001) surface of silicon. To achieve our goal, the Au-induced reconstructed Si(001) and subsequently buffered with an ultra-thin Au layer was used as substrate. The origin of the perpendicular magnetization in the Fe film at both RT and LT cannot be solely treated in terms of film surface/interface anisotropy. Therefore, modifications in the film structure and composition should be considered. This assertion is based on the observation of a transition from the in-plane to perpendicular magnetization upon annealing close to RT in 3 ML thick Fe films grown at LT on Au-covered Si(001). Since such reorientation is thermodynamically prohibited in thin films, thermally driven Au out-diffusion leading to chemical and structural changes in the grown film might be responsible for the observed behaviour. The perpendicular reorientation of magnetization was also observed as the result of capping an annealed 3.2 ML thick Fe film with an ultra-thin Au layer. Thus, we proved that by an appropriate tuning of the growth and annealing temperatures, as well as of the films thickness and sequence, the orientation of magnetization can be rotated out-of-plane in several ML thick films.

The possibility of engineering the magnetization direction even at the ambient temperature in films grown on Si(001) is promising. The films are magnetically 'alive' almost throughout the whole thickness and new perspectives are opened for the possible implementation of the system in spintronic applications.

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