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Large-scale integration of MoS₂ on high-T_C superconducting YBa₂Cu₃O₇ for the realization of Josephson devices

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High- T_c cuprate superconductors' growth conditions and their incompatibility with some of the most standard nanofabrication approaches make their large-scale integration with 2D materials (such as graphene, transition metal dichalcogenides, and other Van der Waals materials) much more difficult than for conventional, metallic superconductors. Here we address this challenge and develop an approach based on pulsed laser deposition that allows the growth of the 2D semiconductor MoS₂ on the archetypal high- T_c superconductor YBa₂Cu₃O_{7-x} (YBCO). This yields functional heterostructures in which the individual constituents' properties are preserved and that show superconductor co-integration with high- T_c superconductors towards the study and leverage of the superconducting proximity effect in hybrid devices.

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The pioneering exfoliation of graphene¹ and the subsequent unveiling of its outstanding electrical properties ² rapidly highlighted opportunities for novel electronic³ and spintronic⁴ devices. Such interest has then quickly been extended to a broader family of 2D materials including, among others, transition metal dichalcogenides (TMDCs)⁵, Xenes⁶, or 2D oxides⁷. Thanks to their unique "surface-like" behavior, a widely explored strategy has consisted in fostering and exploiting proximity-coupling and interfacial effects by combining 2D materials with others in heterostructures, towards discovering new phenomena and applications. One of the prototypical examples is the combination of 2D materials with superconductors. The physics of these hybrid heterostructures attracts much interest for fundamental and technological reasons. This is well illustrated by the use of 2D materials as the weak link of Josephson junctions, where specificities such as the Dirac band structure result in novel physics⁸ and functionalities: for example, gate-tunable Josephson effects⁹ for reconfigurable superconducting electronics¹⁰.

Most of the work in this field has been based on conventional (*s-wave*) low critical temperature (T_C) superconductors, combined e.g. with graphene^{9,11}, topological insulators (TIS)^{12,13}, and TMDCs like MoS_2^{14-17} . However, extensions of those studies to unconventional (*d-wave*) high- T_C cuprate superconductors (HTS) have remained much scarcer, despite their fundamental appeal and technological potential ^{18–20}. Indeed, combining 2D materials with HTS in heterostructures is challenging from a materials science standpoint. On one hand, HTS require epitaxial growth on specific substrates, at high temperatures (hundreds of °C), and in an oxygen-rich atmosphere. This has hampered the standard approach of direct growth onto materials such as graphene or TDCMs flakes conventionally used with low-Tc metallic superconductors ^{9,15–17,21}. On the other hand, the transfer of 2D materials onto HTS is also problematic because these complex oxides are relatively delicate materials: their oxygen stoichiometry (which controls their physical properties) is extremely sensitive to the after-growth handling conditions (temperature, oxygen partial pressure, humidity)²². All of that imposes strong constraints. While a few experiments have surmounted the challenge and fabricated proximity devices that combine HTS with graphene^{23–25} or a few different topological insulators (TIs)²⁶, this was only achieved via transfer techniques and, overall, the accessible heterostructures and fabrication methods

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of thin films of HTS such as YBa₂Cu₃O_{7,x} (YBCO)^{27,28} and many other correlated oxides. Recently, it has also been used to grow individual TMDCs monolayers^{29,30}. We demonstrate here its potential for the growth of HTS/TMDC heterostructures using an archetypal combination: MoS₂ grown on YBCO. As shown below, a critical aspect of this integration is the preservation of YBCO stoichiometry during MoS₂ growth. This is illustrated by varying the MoS₂ growth conditions and monitoring how they affect the superconducting T_c of YBCO. Finally, the functional properties of the optimized HTS/TMDC stack are demonstrated –beyond the characterization of the individual layers– by achieving Josephson coupling across the MoS₂ in vertical heterostructures. The obtained results pave the way for exploring proximity and interfacial effects in a large library of HTS/TMDC heterostructures and lift a barrier towards their large-scale exploitation in superconducting devices and circuits. In the first fabrication step, a c-axis YBCO (50 nm-thick) film is grown on (001) oriented SrTiO₃ (STO) covered with a 6 nm-thick (001) CeO₂ buffer layer. This is done using a PLD system with KrF excimer laser (λ =248 nm), with 94mJ pulses at a repetition rate of 2Hz. The stoichiometric YBCO

are relatively limited as compared to the case of conventional superconductors. This motivates the development of approaches for integrating HTS and 2D materials in heterostructures, preserving their individual physical properties and yielding interfaces of good quality. Ideally, such integration should

Here we show that such large-scale integration can be entirely realized without relying on

transfer techniques, by using instead pulsed laser deposition (PLD). PLD is widely used for the growth

be done over large scales, to facilitate its technological exploitation.

(STO) covered with a 6 nm-thick (001) CeO₂ buffer layer. This is done using a PLD system with KrF excimer laser ($\lambda = 248$ nm), with 94mJ pulses at a repetition rate of 2Hz. The stoichiometric YBCO target is pre-ablated before deposition, which takes place under an O₂ pressure of 0.36 mbar at T=670°C (temperature of the rotating sample holder). After growth, the oxygen pressure is raised to 700 mbar and kept at this level during cooldown, to obtain optimal oxygenation. Once at room temperature, a 10 nm-thick Au capping layer, which protects the YBCO surface, is deposited *in situ* and yields highly-transparent Au/YCBO interface, as well as improves the electrical contact with other materials deposited on top [25,26,33]. The growth of MoS₂ is carried out in a second step, using a PLD chamber equipped with a tripled frequency Nd:YAG (355nm) laser. Several growth conditions were tested to optimize MoS₂ growth while preserving the superconducting properties of YBCO. We varied the three parameters

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 (10^{-1} mbar) , and the deposition is carried out (stoichiometric MoS₂ target, 60mJ laser pulses at a repetition frequency of 2.5 Hz) for a time that determines the MoS₂ thickness. After deposition and before cooldown, the same atmosphere (Ar or O₂) as for warm-up is set in the chamber. Figures 1b, c, and d show the normalized resistance vs. temperature for samples in which MoS2 is grown under different conditions on the YBCO/STO substrate, to illustrate the impact on the superconducting properties of YBCO. The measurements were carried out using a 4-probe configuration by contacting the corners of the samples, the resistance was calculated as R = (V(I) - V(-I))/2I, with $I = 10\mu A$ the injected current and V the measured voltage. Figure 1b illustrates the influence of the presence/absence of oxygen during the temperature ramps (T-ramps): the sample grown in the absence O₂ during the T-ramps shows depressed superconductivity, with a wide, incomplete transition towards zero-resistance whose onset is ~30 K lower than for the sample for which O₂ is present (here T_c^{YBCO} ~ 75 K). This suggests that samples warmed/cooled in Ar are oxygen-depleted. Figure 1c shows resistance versus temperature characteristics R(T) for three samples in which MoS₂ has been grown with different $T_{growth} = 255^{\circ}$ C, 310°C, and 430°C but otherwise identical conditions (O₂ was applied during the T-ramps and the deposition time was 5 min). Here $T_{growth} = 255^{\circ}C$ corresponds to the lowest growth temperature compatible with good MoS2 properties and 430°C corresponds to previously reported growth temperature ~400°C used for TMDCs^{29,30}. One can see that when T_{growth} is increased the superconducting properties are rapidly suppressed. Already for $T_{growth} = 310^{\circ}$ C there is no trace left of the superconducting transition: the resistance shows a metallic behavior likely dominated by the Au layer. For $T_{growth} = 430^{\circ}$ C, the R(T) shows insulating behavior as expected for severely oxygendepleted YBCO³¹. The fact that this is probed indicates that the Au layer is no longer shunting the electrical current, presumably because Tgrowth is high enough to promote Au atom diffusion and segregation, leading to a discontinuity of the Au layer. Figure 1d shows R(T) for three samples grown in identical conditions ($T_{growth} = 255^{\circ}$ C and O₂ during the T-ramps) but with different MoS₂ deposition

shown in **Figure 1a**: the growth temperature, the deposition time, and the presence of oxygen during the warm-up and cooldown ramps. Either a pure Ar (10^{-1} mbar) or O₂ (300 mbar) atmosphere was set during warm-up. Once the growth temperature T_{growth} is reached, the chamber atmosphere is set to Ar

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times. All three show a superconducting transition. However, the T_C decreases with the deposition time suggesting a progressive de-oxygenation of YBCO when heated in pure Ar atmosphere during the MoS₂ growth, even for T_{growth} as low as 255°C. Unfortunately, it is not possible to maintain the O₂ during the growth because oxygen atoms partially replace sulfur, degrading the MoS₂ layer. Hence, to protect as much as possible the YBCO properties, the MoS₂ deposition must be restricted to short times, which as shown below is not a strong constraint if we aim to grow a very thin MoS_2 . Based on $T_{growth} = 255$ °C that allowed protecting at best the YBCO properties, we fabricated heterostructures with different MoS₂ thickness and characterized their structural properties. Fig. 2a shows a typical X-ray diffraction pattern that demonstrates textured YBCO growth along the (001) axis³². Because of their very close scattering vector and due to the peak broadening caused by the finite thickness, the (002) reflection of MoS₂³³ appears convoluted with that of YBCO. This is evidenced by the doubling of this peak's width as compared the YBCO (001) one. The absence of other MoS₂ reflections confirms textured growth along its (001) crystallographic direction. Atomic Force Microscopy (see example in Fig. 2b) was carried out on two areas of the samples, the one being the bare YBCO/Au surface, the other being covered by MoS₂ (the areas were defined by locally etching MoS₂ via oxygen plasma). The root-meansquare surface roughness of MoS₂ σ_{MoS_2} ~3.6 nm, is slightly higher than that of Au σ_{Au} ~3 nm. Measurements of the steps height between both areas (see Fig. 2b) in a series of MoS_2 layers with different deposition times allowed us to estimate the growth rate 7.2±0.8 nm min⁻¹. To further ascertain the quality of the MoS₂, we used Raman spectroscopy³⁴. Typical spectra for MoS₂ and Au surface areas are shown in Figure 2c, taken by using a 514 nm laser, a 3000l/mm grating, under 5mW illumination for 10s. The spectrum measured on the MoS₂ area (blue) shows two peaks at 383.5cm⁻¹ and at 406cm⁻¹ which are absent in the control area (red spectrum). Those peaks correspond to the characteristic E_{2g}^1 and A_{1g} Raman shifts for MoS₂³⁴. To evaluate the sample homogeneity, we looked at the variations of the A_{1g}/E_{2g}^1 ratio, plotted in Figure 2b as a function of the x-y coordinates over a 6 μ m x 6 μ m area. The ratio is nearly constant, with small variations around $A_{1g}/E_{2g}^1 \sim 4.7$, which indicates the homogeneity of the MoS_2 layer over the probed length scale. All of the above confirms the good structural properties of the grown heterostructures.

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conditions make its growth on MoS₂ extremely challenging. In particular, we use UV photolithography (see details elsewhere^{35,36}) to define micrometric square apertures in a resist layer deposited on top of the MoS₂. Subsequently, a 100 nm thick layer of MoSi capped with 50 nm of Au was deposited by sputtering. This way, MoSi contacts the MoS₂ surface only through the square aperture in the resist (indicated with an arrow in the microscopy image shown in Fig. 3a), yielding junctions with an area in the 10 µm x 10 µm range. Four-probe electrical measurements of the junctions were then carried out by directly wire-bonding the YBCO and the MoSi electrodes as sketched in Fig. 3b. Current-biased V(I)and differential conductance dI/dV measurements were performed using the delta-mode of a Keithley 6221 current source coupled to a 2182 nanovoltmeter. Figure 3c presents typical dI/dV vs. I for temperatures 2 K $\leq T \leq 10$ K below the $T_C^{YBCO} \sim 85 \text{ K}$ for this device. The curves are essentially symmetric around zero bias. For T = 10 K > 10 K T_C^{MOSi} , we observe high conductance (~10⁴ S) at low bias, followed by a gradual decrease (down to ~ 300 S) as the bias is increased. The behavior dramatically changes around $T = 5 \text{ K} \sim T_C^{MoSi}$. Already at T = 5 K the low-bias conductance is significantly lower (~10³ S) than above, resulting in a flatter curve. As the temperature is further decreased to T = 4.9 - 4.7 K, the low-bias conductance rapidly drops to the tens of S level. Indeed, within this temperature range the curve's shape is inverted: a "dip" appears within the low-bias range, followed by a sudden increase with increasing bias, ultimately leading to a similar conductance level as for higher temperatures. For $T \le 4$ K, the low-conductance level extends over all the probed bias range, except around zero bias. Indeed, for T < 4.7 K a zero-bias peak develops which becomes higher as the temperature becomes lower, especially below 3 K. This peak results from the transition to a zero-resistance state, as it can be deduced from the corresponding V(I) shown in the main panel of Fig. 3d. One can see that, at the lowest temperatures and within a low-bias range, current

To demonstrate the functional properties of the YBCO/MoS2 heterostructures, and to

characterize the electrical properties of MoS₂, we fabricated YBCO/MoS₂/Mo₈₀Si₂₀ vertical Josephson junctions (as sketched in **Figure 3b**). Here Mo₈₀Si₂₀ (MoSi), which is an amorphous *s*-wave superconductor with $T_c^{MoSi} \sim 5 K - 6 K$ [30], is conveniently used as a top electrode because it can be easily implemented via a simple lithography approach, contrary to YBCO whose restrictive growth

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barrier. The temperature-dependent conductance features described above can be explained as follows. For $T_c^{MoSi} < T = 10 K < T_c^{YBCO}$, the bias dependence is the one typically observed in finitetransparency junctions between a normal metal and a *d-wave* superconductor, which typically display a high conductance at low bias followed by a strong decrease with increasing bias^{37,38}. This behavior may result from two mechanisms: the first one is tunneling into characteristic zero-energy quasiparticlebound states at the surface of the *d-wave* superconductor³⁹. The second is the current-driven depression of superconductivity near the interface. Both mechanisms can be concomitant. The radically different behavior for T < 5 K is associated with the onset of superconductivity in MoSi. The strong conductance drop within the low-bias range is as expected from the energy gap opening in a conventional (*s-wave*) superconductor, which leads to a vanishing density of electronic states around the Fermi level. Here, thermally excited Bogoliubov quasiparticles are the only available states, whose population diminishes as the temperature is lowered, largely hindering single-particle

injection leads to zero voltage drop. This allows defining a superconducting critical current $I_c \approx 200 \ \mu A$ for the measurement at 2 K. I_c quickly vanishes as the temperature is increased, as finely evidenced by the gradual disappearance of the zero-bias conductance peak in the differential conductance, see **Figure 3c**. The *V*(*I*) for another junction is shown in the inset of **Fig. 3d** and displays a similar behavior with

The inset of **Fig. 3c** shows direct conductance I/V measured at high I=40 mA and low T=2 K

for ~20 different junctions with either 0, ~5.6 nm or ~15.5 nm thick MoS_2 . This conductance corresponds high-current limit "plateau" observed in the main panel of **Fig. 3c**. One can see that the conductance decreases exponentially as the MoS_2 thickness increases, as expected for MoS_2 behaving as a tunneling

 $I_C \approx 110 \ \mu A$ at T=2 K.

states, whose population diminishes as the temperature is lowered, largely hindering single-particle tunneling into the superconductor⁴⁰. That explains the strong conductance drop over a bias range that gradually extends as the temperature is decreased (Fig. 3d). The quasiparticle current across the junction is thus strongly suppressed as MoSi turns superconducting. This, together with the behavior observed in the inset of Fig. 3c, rules out the junctions' conductance is dominated by pinholes and supports that

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 MoS_2 behaves as a tunneling barrier. While quasiparticle tunneling is limited upon opening of the superconducting gap in MoSi, Cooper pairs can flow across the device as long as the current is kept below a certain critical I_C (**Fig. 3d**), yielding the emergent zero-bias conductance peak.

A quantitative analysis of the transport measurements would likely require adapting existing models. e.g. for tunneling ³⁹ and Josephson effects ⁴¹ in d-wave superconductor junctions, to our junctions' specificities. While this is beyond the scope of the word, from the above observations, a picture emerges. It is clear from the inset of **Fig. 3c** that, in the presence of a MoS₂ layer, the junctions' conductance is not dominated by the YBCO/Au interface, but instead by the MoSi/MoS₂ one and MoS₂ layer, which behave as a tunneling barrier. Indeed, when MoSi is in its normal state, the bias-dependent conductance reflects characteristics of electron tunneling from this electrode into the "*d-wave* stack". The latter includes YBCO, the thin Au capping (which must be fully proximitized considering its thickness and the transparent interface expected from in-situ deposition^{23,24,42}), and maybe part of the MoS₂ layer which, based on experiments on this material in contact with metallic superconductors¹⁵, could be at least partially proximitized. As MoSi enters its superconducting state, the opening of the energy gap hinders single-particle injection, yielding the conductance drop for finite bias. However, at sufficiently low temperatures, Josephson coupling between the "*d-wave* stack" and MoSi is achieved, and a supercurrent (Cooper pair) flow is observed.

In summary, we have presented here the conditions for the PLD growth and integration of the 2D semiconductor MoS_2 with the high- T_C oxide superconductor YBCO in functional heterostructures. To illustrate the developed approach's potential, we have fabricated superconducting junctions that display Josephson coupling between YBCO and a conventional superconductor (MoSi) across MoS_2 . This realization paves the way to exploring proximity behavior between HTS and TMDCs in its many facets (doping dependence, length scales, ...) via 2D material gating, a property unavailable yet, as well as leveraging them for applications. Because the growth of high- T_C oxides on TMDCs appears an extremely challenging endeavor given their very restrictive growth conditions, the realization of those prospects will likely require implementing the PLD approach to fabricate planar proximity devices with the superconductor at the bottom^{23,24,42}. That constitutes the next step towards gate-tunable high- T_C

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superconducting large-scale devices (superconducting quantum interferometers, SQIFs and similar

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arrays used in applications⁴³) based on TMDCs and HTS.

101130224 "JOSEPHINE", as well as the COST action "SUPERQUMAP".

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Figure 1 (a) Schematic representation of the parameters (temperature during deposition T_{growth} , oxygen pressure P_{O_2} during temperature ramps, and deposition time) used to control the growth of MoS₂ on the Au/YBCO//STO heterostructure. (b)-(d) Comparison of R(T) characteristics for samples grown under varying conditions of (b) P_{O_2} , (c) temperature during deposition T_{growth} (d) deposition time; as indicated in the legends. R(T) measurements are normalized to the resistance at T=300 K.

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Figure 2 (a) X-ray diffraction spectrum (taken with $\lambda_{Cu}=1.5418$ Å) of a MoS₂ (20 nm)/Au (10 nm)/YBCO (50 nm) heterostructure on a CeO₂/STO substrate. The legends identify the reflection peaks. (b) Atomic force microscopy of a MoS₂ (5.4 nm)/Au (10 nm)/YBCO (50 nm) sample .On the right part of the image (darker area) the MoS₂ has been etched, exposing the Au surface. The inset shows a typical topography profile (line scan) across the boundary between both areas, the different heights at left and right sides being equal to the thickness of MoS₂. The white stripe corresponds to a ridge formed by the lithography and etching process (c) Raman spectra taken over Au/YBCO//STO (red) and MoS₂/Au/YBCO//STO (blue) from 280 to 460cm⁻¹. The characteristic A_{1g} and E_{2g}^1 peaks of MoS₂ are identified. (b) 2D map depicting the ratio between the A_{1g} and E_{2g}^1 peaks amplitude over a 6 µm x 6 µm area.



Figure 3 (a) Optical microscope image (top view) and (b) cross-section scheme of one of the studied Josephson junctions. The conductance of the device is measured by injecting current *I* and measuring voltage *V* in a four-point configuration as illustrated. (c) differential conductance of a $10 \,\mu m \ge 10 \,\mu m$ MoSi(100nm)/MoS₂(5.6nm)/Au(10nm)/YBCO(50nm) junction at various temperatures ranging from 10 to 2K. The inset shows the conductance at *I*=40 mA at *T*=2 K for a series of junctions with different MoS₂ thickness t_{MoS2} . Black crosses are the values for individual junctions (around 6 per each t_{MoS2}) and red dots are their average. (d) V(I) characteristic of heterojunction. The inset of (d) shows the V(I) characteristics of a smaller junction (7.5 μ m x 7.5 μ m).

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