Magnetic flux channelling in YBa₂Cu₃O_{7- δ} films by a chemical solution deposition grown technique on vicinal and non-vicinal substrates

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Abstract

Magneto-optical imaging of $YBa_2Cu_3O_{7-\delta}$ films with high critical current density, synthesized by a cost-effective metal organic decomposition technique reveals inhomogeneous flux penetration in the specimens in the form of thin parallel lines. The origin of such a stripy pattern and its dependence on the sample preparation conditions and state of substrate is discussed. The stripes reflect accumulation of planar defects forming parallel lines of reduced in-plane critical current density, j_c , perpendicular to planar defects and enhanced j_c parallel to them. Such channel-like reduction and corresponding enhancement of j_c is especially expressed in a sample deposited on vicinal substrate, which, as a consequence, demonstrates global temperature-dependent in-plane anisotropy with an anisotropy ratio up to 2.4. The directional enhancement of critical current density due to planar defects could be beneficial for practical use of superconducting films. Copyright © 2017 VBRI Press.

Keywords: Magneto-optical imaging, superconducting films, YBCO, metal-organic deposition.

Introduction

Advanced superconducting materials are of primary importance for the development of renewable energy economy [1]. Especially attractive is the use of superconducting materials for simultaneous delivery of fuel (liquid hydrogen) and loss-free electricity [2]. The superconducting material YBa₂Cu₃O_{7-δ} (YBCO) could be sprayed in a solution on curved surfaces and subsequently thermally treated to produce superconducting cover on a specially prepared surface of pipes intended to carry liquid hydrogen. The advantage of YBCO compared, for example, to MgB₂, which is currently the preferred material in suggested modern concepts of such delivery [1, 2], would be less stringent requirements for thermal insulation, since YBCO has much higher critical temperature than MgB₂. Following this concept, in the present work we test superconducting properties of

substrates do not

YBCO films prepared by a spin coating technique, which could be used for covering the pipes of a superconducting grid.

We use magneto-optical imaging (MOI) as a main characterisation technique. It allows obtaining live images of magnetic flux penetrating into the sample [3]. We can obtain local information from superconducting films, clarifying how micro and nano defects influence j_c , which is not possible with other techniques such as transport and magnetisation measurements. This technique allows clarifying why different samples grown on similar necessarily display similar superconducting properties. Films of YBCO have been extensively studied by MOI, see, for instance, [3, 4] and references therein. YBCO films synthesized by metal organic deposition (MOD) have different microstructural properties from those synthesized by the more common pulsed laser deposition (PLD) technique [5], and it is therefore important to understand magnetic flux behaviour in such films.

A layered superconducting material such as YBCO grown on a vicinal substrate can produce a film with an improved critical current density (j_c) in one spatial direction. This results in an in-plane anisotropy, which can be defined as a ratio of the current density in two perpendicular directions in the basal ab-plane of the compound. A vicinal substrate is a substrate that is cut at an angle to a low-index crystallographic plane, resulting in a step-like surface with a few nanometres distance between the steps. When a layered superconductor, such as YBCO or MgB₂, is deposited on such a substrate, epitaxial growth results in a superconducting film with its c-axis oriented at an angle from the normal to the plane, and the film itself may adopt such a step-like structure. In films on vicinal substrates, the stress due to the lattice mismatch between the crystal lattice parameters of the superconductor and substrate relaxes by forming planar defects such as twin boundaries, antiphase boundaries (APBs) or edge dislocations along the miscut steps [6]. Thus, in vicinal epitaxial films, there is a set of parallel planar defects, which induces the in-plane anisotropy of magnetic and electrical properties of the films affecting their critical current density. Several different vicinal substrates, such as SrTiO₃ [7-11], MgO [12], LaAlO₃ NdGaO₃ [14] and $(LaAlO_3)_{0,3}$ -(LAO) [13], (SrAl_{0.5}Ta_{0.5}O₃)_{0.7} [6], have been used for growing YBCO with planar defects.

Previous studies combining MOI, transport measurements and structural characterisation of YBCO have showed that magnetic flux tends to penetrate the sample in the form of one-dimensional lines parallel to the planar defects, and that j_c is decreased perpendicular to these defects and increased parallel to them [7]. The increased j_c is caused by magnetic vortices being pinned by the planar defects. Hence, this is the source of the j_c anisotropy reflected in specific penetration of a magnetic field seen in MOI. The distance between planar defects depends on the miscut angle of the vicinal substrate. The anisotropy ratio of such a sample is defined in MOI as Γ = tan α = j_{CL}/j_{CT} , where α is the angle between a sample's edge and a discontinuity line (d-line) emerging from its corner, and j_{cL} and j_{cT} are critical current densities along and perpendicular to the steps of the substrate, respectively. The anisotropy ratio in a superconductor grown on a vicinal substrate depends on the film thickness [8], temperature [8,15], applied magnetic field [9], and the substrate miscut angle [10]. j_{cL} on the order of the superconductor depairing limit in films grown on vicinal substrates have been reported for YBCO grown on 10° vicinal SrTiO₃ substrates [11]. Tafuri et al., however, have reported that a miscut angle as large as 45° results in films displaying Josephson properties [16].

The mechanism of stress relaxation is different in samples deposited on non-vicinal substrates. They demonstrate various sets of weak boundaries with differently enhanced or reduced j_c . For instance, twin boundaries are commonly found in standard LAO substrates, because of the orthorhombic crystal structure [17], and these are transferred to the grown YBCO film.

In this work, YBCO films deposited on both standard and vicinal (001) LAO substrates were investigated. The films were prepared by a chemical solution deposition (CSD) technique, which is a simple method for preparing high-quality films [18]. One of the solutions we used is completely free from fluorine, which makes the synthesis less harmful to the environment than the standard method that uses a trifluoroacetate (TFA) solution. In earlier works using fluorine-free MOD techniques, the critical current density was significantly lower than in films prepared by PLD [19]. There is a significant progress over years in improving j_c in films made by low fluorine and fluorine-free MOD, and the values comparable to those in films produced by PLD [8] have been obtained recently [20].

Table 1. The names, average film thickness, type of LAO substrate, and precursor solution technique for the three films discussed in this paper. (LF = low-fluorine, FF = fluorine-free, MOD = metal organic deposition, TFA = trifluoroacetate.)

Sample name	Film thickness (nm)	Substrate	Synthesis method
Sample A	220	Standard	LF MOD with TFA [21]
Sample B	305	Vicinal	LF MOD with TFA [21]
Sample C	210	Standard with twins	FF MOD [20]

Experimental

Materials synthesis

Two films of YBCO were synthesized by a low-fluorine MOD route using a TFA solution [21], and one film of YBCO was synthesized by a fluorine-free MOD route using only propionic acid as solvent [20]. Drops of a solution were deposited on LAO substrates by a spincoating technique. The spinning caused the solution to spread and cover the whole substrate, forming a continuous wet layer. The precursors on the substrates were subsequently heat treated to form quasi-singlecrystalline YBCO films. Results from three different samples are presented in this paper. Samples A and B were synthesized from the same solution, namely TFA, and Sample C was synthesized with the fluorine-free method. Samples A and C were deposited on a standard substrate (supplied by CrysTec GmbH), and Sample B on a vicinal substrate (supplied by MTI in China), which was cut at an angle of 10° from the (001)-plane. The two samples deposited on a standard substrate differ in the chemical solution used and that the substrate used for growing Sample C contained twin boundaries. The MOI of the specimen without twin boundaries (Sample A) is mainly presented to demonstrate how magnetic flux penetrates through a sample without extended planar defects. Table 1 shows names, thickness, type of substrate and precursor solution for all samples used in this work.

Characterisation

The specimens were analysed with MOI at different low temperatures achieved by cooling them in a helium cryostat. A magneto-optical layer (MOL), in our case a ferrite garnet film deposited on a transparent gadolinium gallium garnet substrate, is placed on top of the sample. Such MOLs are made by liquid phase epitaxy [22,23]. and their free surface is coated with a thin aluminium mirror layer. Polarised light is sent onto the MOL and reflected from the mirror layer, which is in contact with the sample, into a crossed analyser. The system makes use of the Faraday effect in the MOL. The Faraday effect used here for imaging magnetic flux penetration into superconductors is an important magneto-optical phenomenon expressed in rotation of the plane of polarisation of light in presence of magnetic field. The rotation angle in magneto-optical crystals we use is linearly proportional to the component of magnetic field parallel to the direction of propagation of light. The thickness of the material, in which the Faraday effect occurs determines the accuracy of the technique, which is in our case about 2-5 micrometres. A magnetic field in the sample parallel to the direction of light propagation through the MOL causes a rotation of the polarised light, thereby allowing light to pass through the analyser and hence form an image of the flux penetrating the sample [3].

Magnetic flux penetration is specific in square or rectangular superconducting thin films, where it follows critical state model [24] and begins in central parts of the sample sides avoiding entering along the so-called discontinuity lines. On these lines current is abruptly changing its direction turning 90° and aligning parallel to the corresponding side of the film. The slope of these lines reflects global anisotropy and is an important instrument to characterise flux penetration in the films. Additionally, to this, magnetic flux penetration is strongly influenced by local defects on micro and nano scales, which can lead to unusual flux patterns that are subject of investigation in this paper.

MOI was performed at different temperatures between 3.7 and 92 K, while all three specimens were zero-field cooled (ZFC) to 20 K. Then an external magnetic field was applied perpendicular to the plane of the film and increased to 85 mT or until full penetration of magnetic flux from the edges of the samples.

The thicknesses of Samples A and C were determined by cross-section scanning electron microscopy, and are approximately 220 and 210 nm, respectively. The thickness of Sample B is 260-350 nm as determined by cross-section transmission electron microscopy, so in calculations we use the average thickness of 305 nm.

X-ray diffraction (XRD), more specifically φ -scans and reciprocal space maps (RSM), was obtained with a high-resolution four-circle diffractometer with Cu K α radiation. It was used for structural characterisation of

both films and substrates [20]. An RSM is an intensity map of reciprocal lattice units calculated from the matching angles in XRD [25].



Fig. 1. MO images of YBCO Sample A, which was grown from a low-fluorine solution on a standard LAO substrate. The film was zero-field-cooled to 20 K and a magnetic field was subsequently applied reaching (a) 17 mT and (b) 78 mT. By measuring the distance the flux front has propagated from the lower edge in a), we find that $j_c = 2.1 \cdot 10^{11} \text{ A/m}^2$.

Results and discussion

Sample A: LF MOD with TFA on a standard substrate

Fig. 1 shows magneto-optical (MO) images of the sample synthesized from a low fluorine solution on a standard substrate with no twins. The sample was ZFC to 20 K and a field was applied perpendicular to the plane of the film and increased to 17 mT (**Fig. 1a**), and then increased further to 78 mT (**Fig. 1b**). The MOI experiment as well as analysis using a vibrating sample magnetometer have proved that this sample has good superconducting properties [**26**].

Using **Fig. 1a**, the following equation for Bean's critical state model was used to calculate the critical current density in the film **[27]**:

$$j_c = \frac{\pi H_a}{d} \Big/ \operatorname{acosh}\left(\frac{w}{w-L}\right),$$

where H_a is the applied magnetic field, d is film thickness, L is visible length of flux penetration from the edge, and w is half of the width of the sample.

The equation is valid for long thin strip, but can be used as an approximation for obtained MO images at low flux penetration distances as in **Fig. 1a**, because far from the corners the flux front is almost a straight line parallel to the sample's edge, as in a strip. For sample A, $\mu_0 H_a$ = 17 mT and d = 220 nm. By measuring w/(w - L) = 1.47from the lower edge, we obtain $j_c = 2.1 \cdot 10^{11}$ A/m² at 20 K, which is a high value.



Fig. 2. (a) An MO image of Sample B, which was grown from a low fluorine solution on a 10° vicinal LAO substrate. The film was cooled in zero magnetic field to 20 K and a magnetic field of 17 mT was applied. (b) The field was further increased to 85 mT. By measuring the distance of flux propagation from the lower edge in (a), we found $j_{cL} = 1.8 \cdot 10^{11}$ A/m² in the lower half of the film. With anisotropy ratios of 1.31 in the left half and 1.66 in the right half of the film, which we estimated from (b), we found $j_{cT} = 1.4 \cdot 10^{11}$ A/m² and $1.1 \cdot 10^{11}$ A/m² at the left and right sides, respectively.

Sample B: LF MOD with TFA on a vicinal substrate

MO images of the YBCO film synthesized from a low fluorine solution on a vicinal substrate show a set of parallel channels of magnetic flux. **Fig. 2a** shows an MO image obtained after ZFC the film to 20 K and applying a magnetic field of 17 mT. Using the same equation as for Sample A with d = 305 nm and $\mu_0 H_a = 17$ mT, and measuring w/(w - L) = 1.31 from the lower edge, we conclude that $j_{cL} = 1.8 \cdot 10^{11}$ A/m² at 20 K. Flux has penetrated further into the film from the upper edge, because of the inhomogeneity of the sample's thickness. There are many large defects in the upper part of the film, so we analyse the lower part, which has a higher j_{cL} than

the upper part. The planar defects result in global anisotropy, which can be found from the slope of the discontinuity lines. In **Fig. 2b** we measured the anisotropy ratio of the film at 85 mT, which is, according to the slope of d-lines, equal to 1.31 and 1.66 for the lower left and right corners of the film, respectively (See **Fig. 3** for details on how it was calculated). Similar to the upper and lower edges, the critical current density is somewhat different at the left and right edges as well, also because of the variation of thickness throughout the film, resulting in different penetration depth for magnetic flux. Using relevant anisotropy ratios one can find j_{cT} in the left and right parts of the film as $j_{cT} = 1.4 \cdot 10^{11}$ A/m² and $1.1 \cdot 10^{11}$ A/m², respectively.

The magnetic flux penetration in the film for three different temperatures is shown in Fig. 3. As seen more clearly in Fig. 2a and to a lesser extent in Figs. 3a and b, there are parallel longitudinal lines similar to those observed in [28] emerging from the left and right edges of the sample, better expressed on the left part. These lines are a collective effect of planar defects such as twin boundaries or APBs having preferential orientation along the longitudinal direction of the film. The dark areas between the longitudinal lines of magnetic flux penetration seen in Figs. 2a, 3a and 3b represent low magnetic flux density. The restricted propagation of magnetic flux in these areas could mean strong pinning. Obviously, one cannot see the individual defects in MO images, because the step-width of the substrate and hence distances between the parallel planar defects are on the order of a few nm, whereas the filamentary flux penetration we observe is on the scale of a few μ m. These collectively formed lines are, as a rule, easy channels for magnetic flux penetration and therefore reduce j_c for the current flowing perpendicularly to them. As a result, the film becomes anisotropic as a whole, reflected in a specific motion of flux front to the red lines schematically shown in Fig. 3.



Fig. 3. (a) An MO image of the vicinal Sample B after zero field cooling (ZFC) to 6 K and the application of a field of 68 mT. For the lower left and right corners, the anisotropy ratio (T) is 2.0 and 2.4, respectively. The two values of Γ were found from the tangent of the angle α shown in the figure. (b) An MO image of the sample after ZFC to 20 K and applying a field of 68 mT with corresponding $\Gamma = 1.3$ and 1.7. (c) An MO image of the sample after ZFC to 72 K and the application of a field of 34 mT. $\Gamma = 1.2$ and 1.6. The white arrows in (b) show the directions of j_c along the steps (j_{cL}) and perpendicular to the steps (j_{CT}).

The temperature dependence of the anisotropy ratio is well expressed in Fig. 3. In Fig. 3a, a field of 68 mT was used. It is seen that the flux penetrates much more easily in the longitudinal than in the transverse direction. The film is not homogeneous, but if one measures the angles (α) between the bottom edge and the discontinuity lines emerging from the lower left and right corners, the anisotropy ratio $\Gamma = j_{CL}/j_{CT}$, which could be calculated as $\Gamma = \tan \alpha$ [29], is equal to 2.0 and 2.4 for the left and right sides, respectively. In Fig. 3b, where the sample was ZFC to 20 K and a field of 68 mT was applied, Γ equals 1.3 and 1.7 for the lower left and right corners. In Fig. 3c, the sample was ZFC to 72 K and a field of 34 mT was subsequently applied. For the same corners $\Gamma = 1.2$ and 1.6. As expected, the magnetic field required for full penetration is much lower at 72 K than at 20 K, and the anisotropy ratio is lower as well. Hence, the anisotropy ratio decreases with increasing temperature for YBCO films grown by metal organic deposition, as it does in YBCO films grown by other techniques [14].

Sample C: FF MOD on a substrate containing twin boundaries

The sample grown from a fluorine-free solution is shown in Fig. 4. Fig. 4a is an optical micrograph of the film. Both horizontal and vertical stripes are seen even optically. Fig. 4b shows an MO image of the sample that was ZFC to 20 K with a subsequently applied magnetic field of 17 mT, and in Fig. 4c the field was increased further to 85 mT. From the latter, one can see that there is no clear-expressed in-plane anisotropy in this sample. By measuring the distance of flux propagation into the film from the upper edge with w/(w - L) = 1.39 in Fig. 4b, and with d = 210 nm and $\mu_0 H_a = 17$ mT, we obtain $j_c = 2.4 \cdot 10^{11} \text{ A/m}^2$. This is a high value for films of YBCO synthesized by fluorine-free CSD techniques. **Table 2** compares j_c values at 20 K in our work with j_c in earlier works [19,30] for films made by fluorine-free CSD techniques. Our value is similar to those in films produced by PLD and high-fluorine TFA, with $j_c = 3.4 \cdot 10^{11} \text{ A/m}^2$ [8] and $1 - 2 \cdot 10^{11} \text{ A/m}^2$ [31], respectively.

Table 2. A comparison of j_c obtained at 20 K in our film made by a fluorine-free chemical solution deposition technique (Sample C) with that in earlier works.

Reference	Year	Substrate	$j_c (10^{10} \text{ A/m}^2)$
Bubendorfer et al. [30]	2003	STO, LAO and MgO	1-2
Cui et al.[19]	2009	LAO	2.5
Sample C in the present paper	2016	Standard LAO with twins	24

While the YBCO film deposited on a vicinal substrate (Sample B) shows weak boundaries in only one direction, the situation can be more complicated in YBCO films deposited on non-vicinal substrates, such as Sample C. With no imposed direction, the sample develops

differently oriented sub-domains of weak boundaries, still linked to the direction of the sample's edges. In Sample C, there are parallel channels emerging from the three out of four sample edges, in different directions. The stripes look similar to those observed in the sample on the vicinal substrate, but follow two perpendicular to the edges directions. There are horizontal lines emerging from the edges on the left-hand and right-hand sides, and there are vertical lines emerging from the lower edge.



Fig. 4. (a) An optical micrograph of the YBCO Sample C grown on a standard substrate, showing vertical and horizontal lines caused by twin boundaries in the LAO substrate. (b) An MO image of the specimen after it was zero field cooled to 20 K and an external magnetic field of 17 mT was subsequently applied perpendicular to the plane of the film. By measuring the distance the flux has penetrated from the upper edge, we obtain $j_c = 2.4 \cdot 10^{11} \text{ A/m}^2$. (c) An MO image obtained after increasing the applied field to 51 mT. **Figs. (b)** and (c) show magnetic flux penetrating through low- j_c boundaries in the film.

If one looks carefully at **Fig. 4c**, one can see traces of horizontal lines in the flux penetrated into the film from the upper edge too. These follow the traces of the horizontal lines in the flux emerging from the left and right edges. In other words, some of the horizontal lines in the upper half of the film go through the whole sample, from the left to the right edge. They are invisible in the dlines coming from the corners, and they are clearer in the magnetic flux coming from the side edges, where they are expected to be well-pronounced, than in the magnetic flux coming from the upper edge, where they are not.

The lines seen in the optical image in **Fig. 4a** belong to the LAO substrate, since the YBCO film is too thin to produce significant optical effects. In contrast, the lines of easy flux penetration in the MO images in **Figs. 4b** and **c** are exclusively from the YBCO film, since the LAO substrate is neither superconducting nor magnetic. As we shall see in the XRD results, there are (100) and (110) twin boundaries in the substrate, which result in twin boundaries and possibly other planar defects in the YBCO film grown on top of it.

An XRD comparison of the samples

Fig. 5 shows YBCO (103) φ -scans for all three samples. A φ -scan is normally used for evaluating the in-plane texture quality. In this study, however, we also

characterise twin boundaries in the films and measure the angle between the twins by closely analysing the φ -scan. The full width at half maximum (FWHM) is approximately 1.4° and 1.3° for the single peak in Sample A and B, respectively. FWHM is approximately 0.64° and 0.61° for the left and right peaks in Sample C, respectively. The XRD peak splitting occurs only in Sample C, which indicates that there are distinctive twin boundaries in this sample and not in Samples A and B.



Fig. 5. (a) A φ -scan of Sample A, which was deposited on a standard, flat substrate (the data is also plotted in [20]). The FWHM in this sample is approximately 1.4°. (b) A φ -scan of Sample B, which was deposited on a vicinal substrate. Its FWHM is approximately 1.3°. (c) A φ -scan of Sample C, which was deposited on a substrate containing twin boundaries (the data is also plotted in [20]). FWHM here is approximately 0.64° and 0.61° for the left and right peaks, respectively. Peak splitting caused by twin boundaries is only present in Sample C.

Figs. 6a and 6b present YBCO (103) reciprocal space maps of Samples A and C, respectively. One can see that more than one concentrated peak is observed in Fig. 6b. This confirms that Sample C has twins in the YBCO film and Sample A does not. From the two RSMs of YBCO (103) peak, one can see an epitaxial relationship between the LAO substrate and the YBCO film. The intense sharp peaks are from the LAO (100) and (110) twins with small misorientation angles, while the peaks at low Q_z positions are from the YBCO (103) and (013) reflections. The concentrated peak intensities are a strong evidence of the formation of sharp in-plane textures on both YBCO films, indicating an excellent epitaxial relationship between the substrate and the film. By closely looking at the two RSMs, however, we notice that peak-splitting of YBCO (103)/(013) occurs in Sample C, while there is no major peak splitting in Sample A. This YBCO peak-splitting in Sample C is partly associated with the superior epitaxial growth of YBCO on the LAO substrates with twins. Namely, YBCO grains nucleate on LAO (100) and (110) twins separately. With the growth proceeding, YBCO domains meet, and a twin boundary forms. In Sample A, however, larger stress is present due to a different growth method, which results in a wide XRD peak. Hence, the differences between the films are due to both the presence of twins in the substrates and different synthesis routes.

o reciprocal space

Fig. 6. (a) A (103) reciprocal space map of Sample A, which was deposited on a standard substrate. (b) A reciprocal space map obtained for Sample C, which was deposited on a substrate containing twins. Twins in the YBCO films are observed in Sample C and not in Sample A, in agreement with **Fig. 5**.

Conclusion

Magneto-optical imaging, φ -scans and reciprocal space mapping experiments were carried out on three YBCO films with high critical current density, prepared by a cost-effective spin-coating technique using low-fluorine trifluoroacetate-based and fluorine-free propionate-based solutions. One of the films was grown on a vicinal LAO substrate and two of them were grown on standard substrates. It was found that two of the films contained multiple channels of low j_c caused by the collective effect of planar defects. The specimen on a vicinal substrate contained weak parallel channels of reduced j_c in one direction only, whereas one of the specimens on a

The nano-scale defects, which are invisible individually in conventional optical and magneto-optical imaging, collectively create in both well-observable patterns, which are evenly distributed along one selected direction in the sample deposited on a vicinal substrate. However, they are also present in the sample deposited by the spincoating technique on one of the standard substrates forming several subsets perpendicular to the edges of the sample. In the latter case, they are initiated by the twin boundaries in the LAO substrate.



standard substrate had such lines in two perpendicular directions. In the latter sample, it was found by XRD φ -scans and reciprocal space mapping that there are multiple well-distinguished twin boundaries in the YBCO film originated from the twins in the LAO substrate. These are absent in the other samples. The sample on a vicinal substrate had certain local areas of very high flux pinning. Planar defects result in flux channelling, but, in the film grown on a vicinal substrate, they also result in global anisotropy with high anisotropy ratio up to 2.4. We see the effect of many small planar defects at a much larger scale. The design of defects on nanoscale would allow enhancing critical current of a superconducting cover in a selected practically important direction.

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Author's contributions

Conceived the plan: TQ, YZ, PM, J-CG, HS; Synthesized the samples: YZ, YX; Performed the experiments: TQ, YZ, PM; Data analysis: TQ, YZ, PM, JIV, THJ; Wrote the paper: TQ, YZ, PM. Authors have no competing financial interests.

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