

Article

Effect of Chlorine Addition on Texture and Superconducting Performance of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ -Coated Conductors

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Abstract: $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) thin films with the addition of Cl were prepared on Hastelloy tape by the extremely low fluorine metal-organic deposition (MOD) technique. The composition and microstructure of the present samples were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was revealed that the Cl-added YBCO thin films achieved better texture and density compared with the pure YBCO films. The superconducting properties at magnetic fields were measured using the magnetic property measurement system (MPMS). The results showed that the sample onset transition temperature ($T_{c, \text{on}}$) did not change significantly after the addition of Cl. The critical current density (J_c) in the presence of YBCO with the addition of Cl increased significantly. Meanwhile, both the J_c and gravitational force (F_p) of the (Cl, Hf) co-added YBCO films were further enhanced at different magnetic fields. The thermodynamic analysis of the BaCO_3 removal reaction revealed that the addition of Cl to YBCO formed chlorine compounds, which were effective in avoiding the formation of BaCO_3 . Further, the calculation results of the Gibbs free energy at different pressure quotients showed that the stability of the relevant chlorine compounds occurred in the order of $\text{BaCl}_2 > \text{CuCl}_2 > \text{YCl}_3$ and the oxidation of BaCl_2 to BaO required a higher temperature compared to BaF_2 , implying the potential advantages of inhibiting the a -axis growth.

Keywords: YBCO film; MOD; Cl added; Gibbs free energy



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1. Introduction

There has been impressive progress made in $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (REBCO, RE = Y, Gd, etc., rare-earth)-coated conductors in recent years, while their in-field current-carrying properties still need to be improved. Building appropriate artificial flux-pinning centers in the coated conductor can significantly improve its current carrying capacity. The methods of manually introducing the pinning centers mainly include substrate surface modification [1,2], the introduction of an impurity phase [3,4], rare-earth doping [5–7] and particle irradiation [8,9]. At our group, Ta ion irradiation and doping with five mixed elements were applied to YBCO-coated conductors to produce columnar defects with the size of 7–9 nm in the films [10], forming effective pinning centers with enhanced flux-pinning properties. However, particle accelerators are not common and the high experimental cost is not conducive to mass production. In contrast, with the MOD method, it is easy to control the elemental composition and facilitate the introduction of impurity phases and it has been widely used. A. Gupta et al. [11] first proposed the use of a fluorine-containing metal salt of trifluoroacetic acid to form an intermediate transition phase of fluoride during pyrolysis treatment, effectively avoiding the production of BaCO_3 , which is the so-called TFA-MOD method. A number of research groups have investigated the replacement of

Y ions in YBCO with rare earth elements such as (Y, RE) BCO by the TFA-MOD, which effectively improves the J_c values in strong magnetic fields [5–7]. C. Cai further revealed that ternary rare-earth (Nd, Eu, Gd)Ba₂Cu₃O_{7- δ} films showed a higher critical current density (J_c) and a larger irreversibility field (H_{irr}) compared to mono-rare earth YBCO films [5].

It is known that superconducting phase stability is increased when replacing oxygen with chlorine [12]. Recently, Takanori Motoki et al. reported [13–15] the preparation of halogen (Cl, Br) added YBCO thin films on single crystal SrTiO₃ by the fluorine-free MOD method led to the internal epitaxial growth of small nanocrystals Ba₂Cu₃O₄Cl₂, which effectively promoted the biaxial orientation of YBCO crystals. Further studies showed that halogen-added YBCO films not only improved the flux-pinning performance of the films, but also expanded the synthesis conditions. However, there are still some research questions that have not been clarified yet. Firstly, the mechanism of the halogen Cl promoting the epitaxial growth of thin films during the nucleation process of YBCO and its intrinsic mechanism are not yet clear. Secondly, Cl and F are also halogen elements with similar chemical properties; whether Cl forms an intermediate transition phase of chlorine compounds to avoid the production of BaCO₃ in the process of YBCO pyrolysis treatment remains to be investigated.

In this study, we have prepared Cl-added YBCO thin films on flexible metal substrates by the extremely low fluorine metal organic deposition (EF-MOD) technique. The results show that the J_c values of the Cl-added YBCO films are significantly higher than those of the Cl-free YBCO film samples. In order to further improve its superconductivity under a magnetic field, Cl and Hf co-added YBCO films were prepared, and the results showed that their field superconductivity was significantly further improved. The Gibbs free energy of the chemical reaction during the pyrolysis process of the “chlorine compound process” was also calculated by thermodynamic analysis to analyze the growth mechanism of the Cl-added precursor YBCO.

2. Experimental Method

In this paper, Cl-added and (Cl, Hf) co-added YBa₂Cu₃O_{7- δ} thin films were prepared by extremely low fluorine MOD with only 23% fluorine content (Ba:F = 1:6). The stoichiometric ratio of the solution was Y:Ba:Cu:Cl:Hf = 1:2.1:3.15:0.1:0.01. Yttrium acetate and copper acetate were dissolved in propionic acid and deionized water; barium acetate was dissolved in trifluoroacetic acid and deionized water. Cl-doping and Hf-doping were introduced through hydrochloric acid and hafnium acetylacetonate, respectively. The final precursor solution with a total cation concentration of 2.5 mol/L was obtained by stirring and multiple distillations. The precursor films were coated by the lifting method on the Hastelloy tape with the buffered layers of Al₂O₃/Y₂O₃/IBAD MgO/EPI MgO/LaMnO₃. Then the precursor film was placed in a tube furnace for low temperature pyrolysis to obtain low-temperature film. These lifting and calcination processes were repeated twice. The medium and high temperature treatment was then performed, with the medium temperature stage held at 520 °C for 60 min, then ramped up to a high temperature of 780 °C and held for 180 min. The medium and high temperature pyrolysis stage was carried out in a humid nitrogen oxygen mixture with an oxygen partial pressure of 150 ppm. The gas flow rate was controlled at about 0.2 L/min and the water pressure was 4.2%. The temperature was cooled down to 450 °C and held in flowing oxygen for 90 min at least. During the process, the heating rate of the high temperature stage was 25 °C/min and the cooling rate was 10 °C/min and the resultant thickness of the presented YBCO film was about 1.2 μ m.

The phase purity of the films and the out-of-plane texture of the YBCO films were characterized by θ -2 θ scan and ω scan mapping of X-ray diffraction. The surface morphology of the films was examined using field emission scanning electron microscopy and the M-T curves and the hysteresis loop M-H curves of the added YBCO films were measured to study the superconductivity properties of the samples by the magnetic measurement

system MPMS (model MPMS 0-9 T Quantum Design). In addition, the critical current density J_c was calculated using the Bean model for the obtained data.

3. Results and Discussion

3.1. Surface Morphology and Texture with Cl and Cl-Free

Figure 1 shows the XRD patterns of two-layered YBCO films, Cl-free, Cl added and (Cl, Hf) co-added. The results showed a strong c -axis orientation in all films. The YBCO (103) peak was detected only in the Cl-free sample, which directly reflects the presence of randomly oriented YBCO grains in the films because the addition of Cl suppresses the generation of randomly oriented grains. The a -axis oriented (100) and (200) diffraction peaks were also observed in the Cl-free sample, indicating that the addition of Cl promotes the c -axis epitaxial growth of the films. A small Y_2O_3 peak was observed in the XRD pattern of the Cl-added YBCO films, especially; however, almost no other impurity phases were detected. The $BaHfO_3$ phase was not detected in the (Cl, Hf) co-added YBCO films due to the relatively small amount of Hf addition.

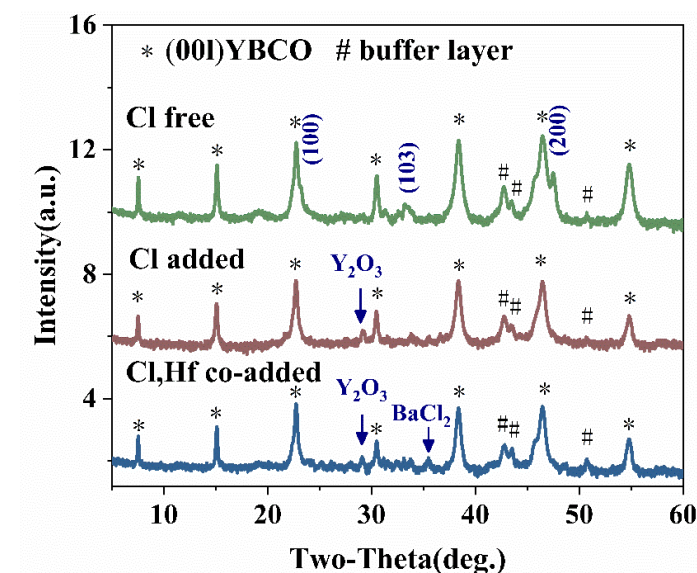


Figure 1. The surface XRD patterns of Cl-free, Cl-added and (Cl, Hf) co-added YBCO films.

To further understand the effect of the Cl addition on the weaving of the YBCO films, the $I(200)/[I(200) + I(005)]$ diffraction intensity ratio of XRD was calculated, and the typical ω scan rocking curves around the (005) peak were performed to estimate the quality of out-of-plan alignment. As shown in Figure 2, the a -axis diffraction intensity ratio of the Cl-added sample was significantly lower and the Cl addition played an important role in suppressing the a -axis-oriented YBCO grains. The full width at half maximum (FWHM) of the (005) peaks were 1.44, 1.08 and 1.12° for Cl-free, Cl-added and (Cl, Hf) co-added sample films, respectively, showing the good out-of-plane alignment. In contrast, the FWHM of the Cl-added sample was the smallest, indicating that the Cl-added sample had a good out-of-plane texture. With the addition of Hf, the out-of-plane texture of the (Cl, Hf) co-added sample films did not deteriorate significantly.

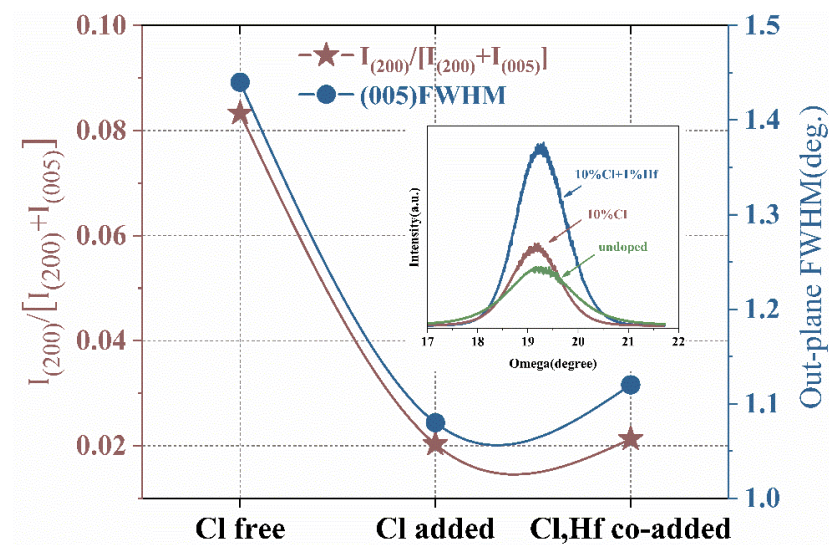


Figure 2. Omega scans of (005) peak for the studied thin films and the (200)/(200) + (005) diffraction intensity ratio.

Figure 3 shows the surface morphology of the studied YBCO films, and it can be seen that the surface of all the film samples was relatively smooth and crack-free. The *a*-axis was clearly visible in the pure YBCO films, while the *a*-axis oriented grains were not observed in the Cl-added and (Cl, Hf) co-added YBCO films. The surface adhesion of the added YBCO films was reduced together with the porosity and the films were denser.

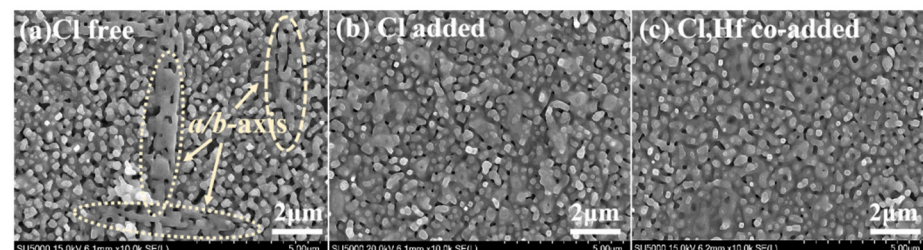


Figure 3. SEM images of Cl-free, Cl-added and (Cl, Hf) co-added YBCO films.

3.2. Superconducting Performance at Various Temperatures and Magnetic Fields

In order to study the superconductivity properties of the samples in the field, we tested the magnetic properties of YBCO films with MPMS. The zero-field-cooled curve of the sample was obtained by applying an external 500 Oe magnetic field. The normalized magnetization intensity versus temperature curves for different YBCO samples are shown in Figure 4. The onset transition temperature of the Cl-added YBCO sample was about 89.5 K, which was slightly larger than that of the Cl-free YBCO sample at 88.6 K. With the addition of Hf, the onset transition temperature of the samples decreased slightly. Overall, the addition of Cl did not much change the superconducting transition temperature of YBCO.

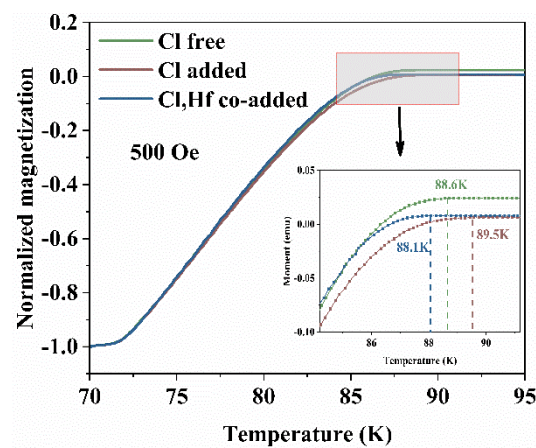


Figure 4. Normalized magnetization intensity curves with temperature for YBCO films Cl-free, Cl-added and (Cl, Hf) co-added.

By applying an external magnetic field parallel to the c-axis direction, the M-H curves of the sample films were measured at 30 K and 77 K, and the critical current density J_c of the samples was calculated by using the Bean model [16], the results of the tests and calculations are shown in Figure 5. The figures show the performance of the added sample at zero field was significantly enhanced due to the increase in the superconducting diamagnetic phase. Furthermore, the presence of the J_c of added samples under different external fields was also enhanced compared to the Cl-free samples, where the presence of the J_c of (Cl, Hf) co-added YBCO films was enhanced more significantly.

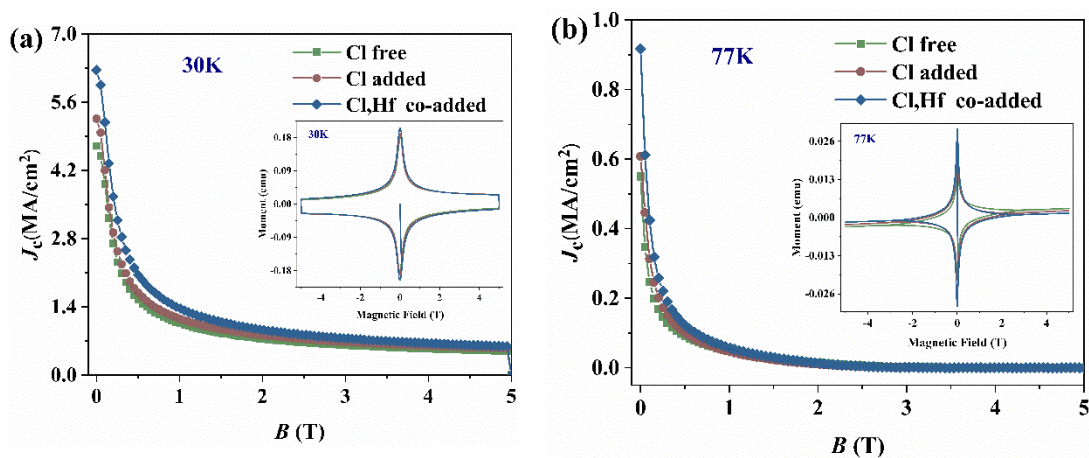


Figure 5. Field dependence of critical current density at 30 K and 77 K for Cl-free, Cl-added and (Cl, Hf) co-added YBCO films. (a) 30 K. (b) 77 K.

To further investigate the internal flux-pinning performance of the added samples, the pinning force is usually calculated by using $F_p = J_c \times \mu_0 H$. The results are shown in Figure 6; the pinning force of all samples increased, which indicates that the appropriate amount of Cl addition can not only promote the c-axis orientation epitaxial growth of the film but also improve the pinning force of the YBCO-coated conductor.

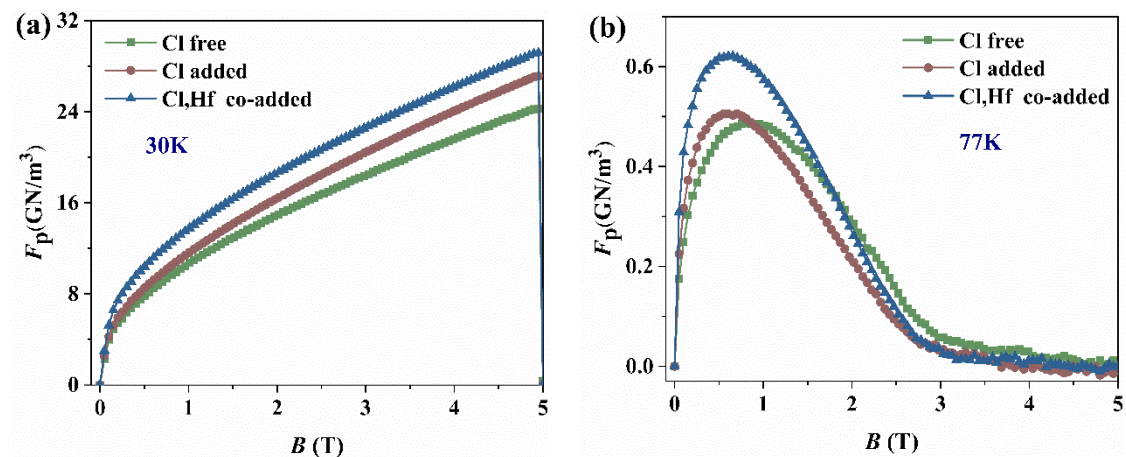
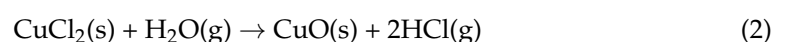
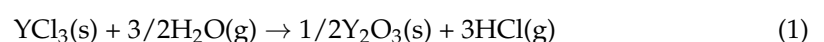


Figure 6. The magnetic pinning force of Cl-free, Cl-added and (Cl, Hf) co-added YBCO samples at (a) 30 K, (b) 77 K.

3.3. Thermodynamic Physicochemical Theoretical Analysis

The nucleation modes of thin films mainly include the uniform nucleation and nonuniform nucleation, where uniform nucleation refers to spontaneous uniform nucleation relying on its own structure, while nonuniform nucleation refers to nonspontaneous, inhomogeneous nucleation induced by an external plasma or substrate. Nonuniform nucleation dominates in YBCO films due to the presence of interfacial energy between the substrate and YBCO [17]. For any deposition method, the nucleation growth process of thin films is quite complex and it includes a series of thermodynamic and kinetic processes. During film formation, the whole system is in a thermodynamically nonequilibrium state, and nonequilibrium systems always have a tendency to transition to the equilibrium state in order to reduce the Gibbs free energy of the system. By calculating the Gibbs free energy, it is possible to further determine the exact direction and extent of the reaction that actually takes place during the heat treatment of YBCO films.

The success of TFA-MOD is based on the BaF_2 mechanism, which can effectively avoid the formation of BaCO_3 [11]. Both element Cl and element F are halogenated elements of the same group with extremely similar chemical properties. The Cl ions were introduced into the YBCO precursor solution to form the corresponding chlorine compounds, and calculations using pure substance thermochemical data revealed that the chlorine compounds can also serve to eliminate BaCO_3 . As shown in Figure 7a, the standard Gibbs free energies for the reaction of fluoride and chloride with BaCO_3 were calculated separately. If BaCO_3 is present in the solution, the system is unstable, and CuCl_2 or YCl_3 will react with BaCO_3 as well as fluoride to form BaCl_2 and the corresponding oxides, effectively circumventing the formation of BaCO_3 . However, the difference is that CuF_2 can further react with Y_2O_3 to form copper oxide and yttrium fluoride with stability $\text{BaF}_2 > \text{YF}_3 > \text{CuF}_2$, as shown in Figure 7b, while the reaction between CuCl_2 and Y_2O_3 is the reverse; CuO reacts with YCl_3 to form copper chloride and yttrium oxide, and the final stability is $\text{BaCl}_2 > \text{CuCl}_2 > \text{YCl}_3$. As the temperature increases, chlorides react with water vapor to form the corresponding oxides:



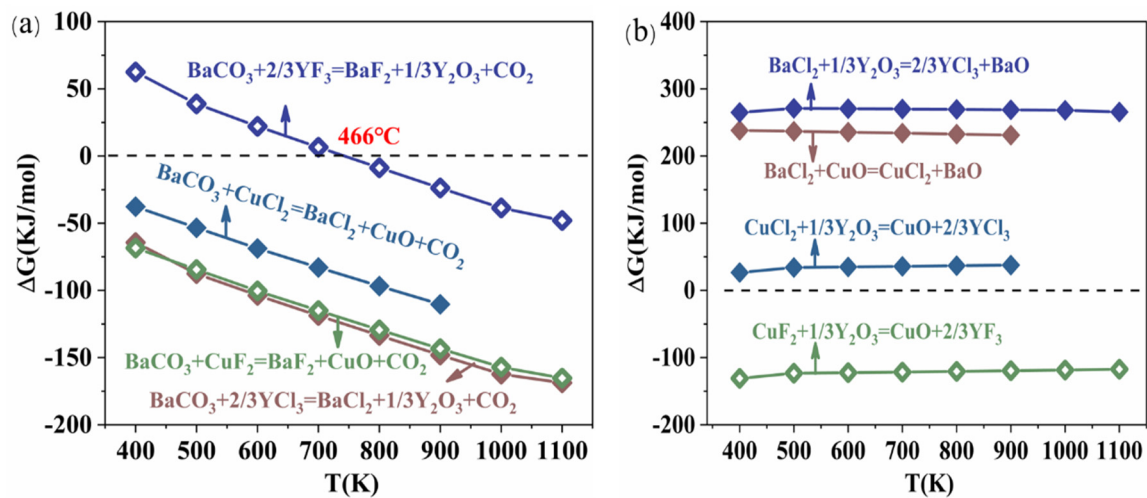


Figure 7. Gibbs free energy of each reaction formula: (a) Gibbs free energy change of BaCO_3 removal reaction; (b) further reaction between products.

The free energy of the entire reaction varies with the activity of the reactants and products. The magnitude of the proportional relationship between the partial pressure of the released gas and the water pressure during the heat treatment also affects the direction of the reaction proceeding. The Gibbs free energy change of the reaction process can be expressed as [18]

$$\Delta G_T = \Delta G_T^\theta + RT \ln J_P \quad (3)$$

where J_P is the pressure quotient, let $J_0 = P_{\text{HCl}}^2 / P_{\text{H}_2\text{O}}$, then the J_0 values of chemical reaction Equations (1) and (2) are J_0^3 and J_0 , respectively. The Gibbs free energy changes of the reaction Equations (1) and (2) are plotted for three different pressure quotients $J_0 = 1$, 1×10^{-8} , 1×10^{-13} [19], as shown in Figure 8. As the partial pressure of HCl decreases gradually, the free energy is driven below the negative value and the reaction will proceed from left to right.

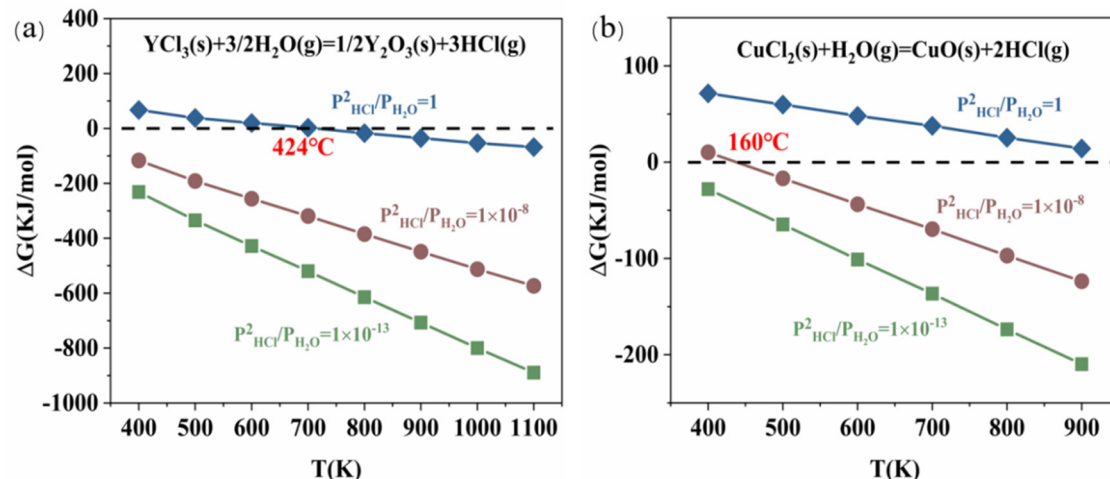
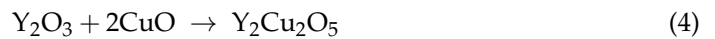


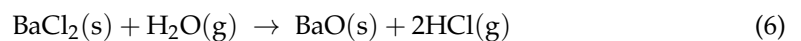
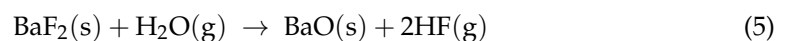
Figure 8. Gibbs free energy changes under different proportions of exhaust gas partial pressure and water pressure: (a) YCl_3 reacts with water vapor; (b) reaction of CuCl_2 with water vapor.

The Gibbs free energy value for the reaction of YCl_3 with water starts to be less than zero near 424°C at $J_0 = 1$, which indicates that reaction Equation (1) proceeds to the right, while the Gibbs free energy value for the reaction of CuCl_2 with water remains greater than zero at high temperatures. With the discharge of HCl gas, the value of J_0 decreases

to 1×10^{-8} , as shown in Figure 8b, the Gibbs free energy of reaction (2) starts to be less than zero at lower temperatures, the reaction proceeds to the right to form CuO and HCl. The thermodynamic point of view shows that YCl_3 in the precursor solution reacts with water preferentially to form Y_2O_3 , and as the partial pressure of HCl decreases CuCl_2 reacts with water to form CuO. Y_2O_3 is formed first before CuO, which effectively prevents CuO grains from growing too large, and plays a role in suppressing random grain orientation. The generated oxide continues to react:



Based on $\Delta G(\text{KJ/mol}) = 18.47 - 0.0219T(\text{K})$, reaction (4) proceeds at $>570^\circ\text{C}$ in preparation for subsequent YBCO nucleation [20]. As the temperature continues to increase, barium fluoride or barium chloride is subsequently oxidized to form barium oxide [20]:



The Gibbs free energy changes of the reaction Equations (5) and (6) are plotted separately for three different pressure quotients as shown in Figure 9. At high partial pressures of the exiting gas, the Gibbs free energy values for the reaction of either BaF_2 or BaCl_2 with water are >0 , and the reaction cannot proceed to the right. The Gibbs free energy value for the reaction of BaF_2 with water starts to be less than zero near 657°C when the pressure quotient drops to 1×10^{-13} , and the reaction (5) proceeds to the right, with BaF_2 reacting with water vapor to form BaO. At the same pressure quotient, BaCl_2 needs a higher temperature to react with water, and BaCl_2 reacts with water vapor to form barium oxide at higher than 681°C . Compared with BaF_2 , BaCl_2 oxidizes to BaO at a higher temperature, which effectively inhibits the *a*-axis growth and thus promotes the *c*-axis epitaxial growth of the film. During the actual experimental operation, we could increase the partial pressure of H_2O or use a higher flow rate to reduce the partial pressure of the gas exiting the film surface, thus increasing the driving force of the reaction from BaF_2 or BaCl_2 to BaO. The chemical reaction equation for the final formation of YBCO is:

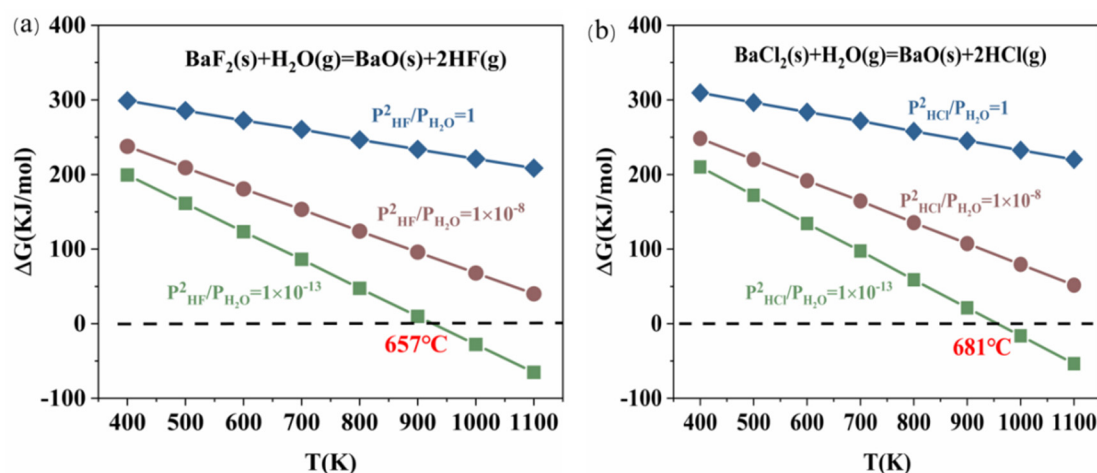
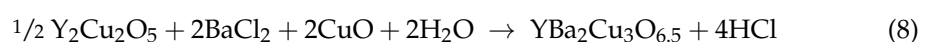
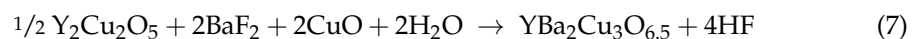
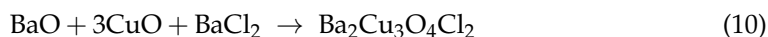


Figure 9. Gibbs free energy changes under different proportions of exhaust gas partial pressure and water pressure: (a) BaF_2 reacts with water vapor; (b) reaction of BaCl_2 with water vapor.

Takanori Motoki and Yui Setoyama epitaxially grew $\text{Ba}_2\text{Cu}_3\text{O}_4\text{Cl}_2$ small nanocrystals with Cl-added YBCO films [13–15]. The reaction equation for the formation of $\text{Ba}_2\text{Cu}_3\text{O}_4\text{Cl}_2$ may be:



The process phases and mechanism of action of chlorine in YBCO are shown in Figure 10. With the addition of 23% fluorine, almost all of the barium formed BaF_2 and a small amount formed BaCl_2 , avoiding the formation of BaCO_3 . In addition, the presence of vapor promoted the formation of HCl and Cl was expelled from the superconductor, so that no $\text{Ba}_2\text{Cu}_3\text{O}_4\text{Cl}_2$ was formed.

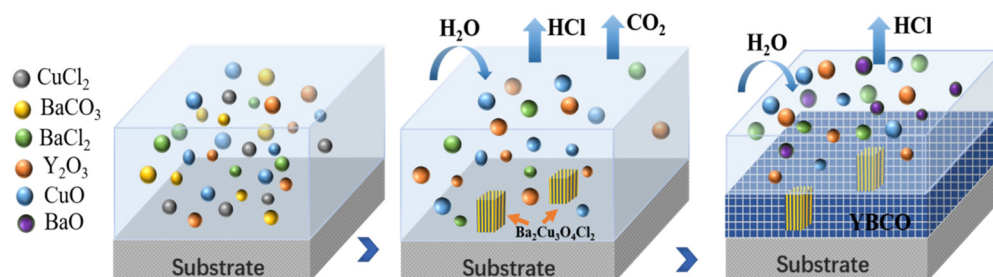


Figure 10. Growth mechanism of Cl-added YBCO grains.

4. Conclusions

In this paper, Cl-added and (Cl, Hf) co-added YBCO films were successfully prepared on a Hastelloy buffer layer by the extremely low fluorine MOD method. The studied YBCO film texture and superconducting properties were investigated. The XRD patterns revealed that the addition of chlorine is able to effectively inhibit the a -axis growth, leading to the better texture performance. The surface morphology for the Cl-added samples appeared more solid and dense, while the critical transition temperature of all the studied samples was basically unchanged. The J_c and pinning force density for the co-addition of Cl and Hf samples were effectively enhanced at different magnetic fields. The change of Gibbs free energy during the reaction of chloride with BaCO_3 was calculated by thermodynamic analysis of the BaCO_3 removal reaction, showing that the formation of chloride compounds by adding Cl in YBCO appear effective to avoid the formation of BaCO_3 . Furthermore, the change of Gibbs free energy during the process of oxidation of BaF_2 or BaCl_2 and formation of BaO was calculated at different pressure quotients, implying that a higher temperature was required for the oxidation of BaCl_2 to BaO compared with BaF_2 , making the MOD application a promising technique to inhibit the a -axis growth.

Author Contributions: Conceptualization, Z.Y. and R.H.; methodology, S.Q.; software, M.C.; validation, J.C., M.C. and Z.L.; formal analysis, M.C.; investigation, M.C., R.H.; resources, C.C.; data curation, M.C. and J.C.; writing—original draft preparation, M.C.; writing—review and editing, M.L., Z.L. and C.C.; visualization, C.C.; supervision, C.C.; project administration, C.C.; funding acquisition, C.C. All authors have read and agreed to the published version of the manuscript.

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