High-throughput research on superconductivity

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TOPICAL REIVEW — Physics research in materials genome

High-throughput research on superconductivity*

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As an essential component of the Materials Genome Initiative aiming to shorten the period of materials research and development, combinatorial synthesis and rapid characterization technologies have been playing a more and more important role in exploring new materials and comprehensively understanding materials properties. In this review, we discuss the advantages of high-throughput experimental techniques in researches on superconductors. The evolution of combinatorial thin-film technology and several high-speed screening devices are briefly introduced. We emphasize the necessity to develop new high-throughput research modes such as a combination of high-throughput techniques and conventional methods.

Keywords: superconductivity, materials genome initiative, high-throughput experimental technology, high-throughput research mode

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1. Introduction

High-temperature superconductors are promising materials for applications in the fields of lossless power transmission, magnetic levitation transport and magnetic resonance imaging to name a few, but the practical applications are limited by the lack of appropriate materials. Therefore, it is of great importance to investigate the mechanisms of high-temperature superconductivity and explore new superconductors with suitable performance parameters such as the transition temperature (T_c) and the critical current density. The essential task of establishing a complete and accurate phase diagram for high-T_c superconductors is faced with two obstacles. First, combining more than a few elements appears to be a promising route for pursuing high-temperature superconductivity, given the fact that the record of $T_c \sim 138$ K at ambient pressure is currently maintained by the six-element superconductor $Hg_{0.8}Tl_{0.2}Ba_2Ca_2Cu_3O_{8.33}$.[1] However, the amount of studies performed on compounds with large number of element is limited. Besides, the properties of high-temperature superconductors are extraordinarily sensitive to the proportion of its constitutive elements.[2,3] For instance, changing the cation doping concentration on the order of 1% in cuprates such as La_2CuO_4 can lead to transitions between insulator, metal, and superconductor.[4] A slight difference in oxygen content significantly impacts the properties of oxide superconductors also.[5] Second, there are many variables such as doping[6] and pressure[7] in the phase diagrams of superconductors. It is difficult to experimentally achieve consistency in some of these synthesis parameters among samples batches, leading to significant uncertainty in the phase diagrams. The inaccuracy of phase boundaries makes it non-trivial to study the phase transition behavior around the critical points.[8] In other words, the multi-element and multi-variable nature of synthesis conditions of unconventional superconductors represents a significant challenge to the efficiency and precision of materials exploration. A new approach is required to change the slow and serendipitous trial-and-error process.[9,10] Fortunately, high-throughput materials synthesis and characterization methodology, which has been developed over the last two decades, has proved to be capable of accelerating materials exploration.[11–15] The high-throughput experimental strategy is considered as one of the three key components of the Materials Genome Initiative (MGI).

What is the genome in the MGI? First, materials "genes"

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represent the elementary units determining the characteristics of a class of materials, *e.g.*, copper–oxygen planes in cuprate superconductors. Second, preparation of materials libraries and their rapid screening are central to the MGI. This is analogous to the antibody preparation technology in biological genetic engineering, where the strongest antibodies are selected and cultured. This strategic methodology launched in 2011 by the former president of the United States, Barack Obama, aims to develop new materials research and development procedures and set up efficient information sharing mechanisms.[16] The MGI activities mainly consist high-throughput computations and synthesis, rapid characterization, and establishing comprehensive material databases.[17] Combined with information technologies such as machine learning, high-throughput calculations and simulations will help to achieve intelligent design and streamlined investigation of new materials. It is believed that credible predictions could be made in a short time[18,19] to guide experiments and increase the probability of discovering desired materials.[20] High-throughput synthesis technologies can be used to continuously change parameters such as materials growth temperature[21] and doping concentration.[22,23] In this way a series of samples can be synthesized in parallel so that the preparation time is significantly shortened. High-throughput characterization methods with high spatial resolution can perform micro-region detection and continuous scanning.[24] Appropriate high-speed screening instruments including *in-situ* characterization of structure, composition, and other physical properties will benefit establishment of materials databases.[25] Accordingly, the implementation of the MGI will shorten the research and development cycle of new materials effectively and save costs. In this manner, high-throughput techniques can benefit the research on superconductivity.

2. An overview of high-throughput methods

As an essential part of the MGI, high-throughput materials synthesis and characterization technology, whose central idea is to move from sequential iteration to parallel processing plays a critical role in transformation from experiential methods to predictive methods. We will briefly summarize the development of combinatorial thin-film growth technologies and rapid characterization methods applied to superconducting thin films. More comprehensive and detailed descriptions of combinatorial approaches are available in other reviews.[10–12,14]

The first report of combinatorial thin films using the co-deposition method, dates back to 1965.[27] Kennedy *et al.* quickly obtained the Fe–Cr–Ni ternary library on an equilateral-triangle metal foil with a side length of 10 inch (1 inch = 2.54 cm) by electron–beam co-evaporation. This technique offers an efficient way to map phase diagrams,[28] which is of key importance to explore new superconductors. In 2013, Jin *et al.* took advantage of the composition spread technique to confirm the superconductivity in the Fe–B binary system.[26] They fabricated by co-sputtering the Fe–B binary compositional phase space across 3-inch Si wafers with a 200-nm SiO_2 layer on top.[29] Figure 1(a) shows the photograph of one composition spread wafer taken under ambient light. The average composition at different solid circle positions is obtained by wavelength dispersive spectroscopy. Besides, the 64-pogo-pin set up with 16 multiplexed channels was used to simultaneously measure the resistance *versus* temperature dependence of 16 spots on the diced 1 cm × 1 cm chips. As can be seen in Fig. 1(b), the sample undergoes a transition from the metallic Fe-rich crystalline region to the semiconducting/insulating B-rich nanocrystalline/amorphous region.

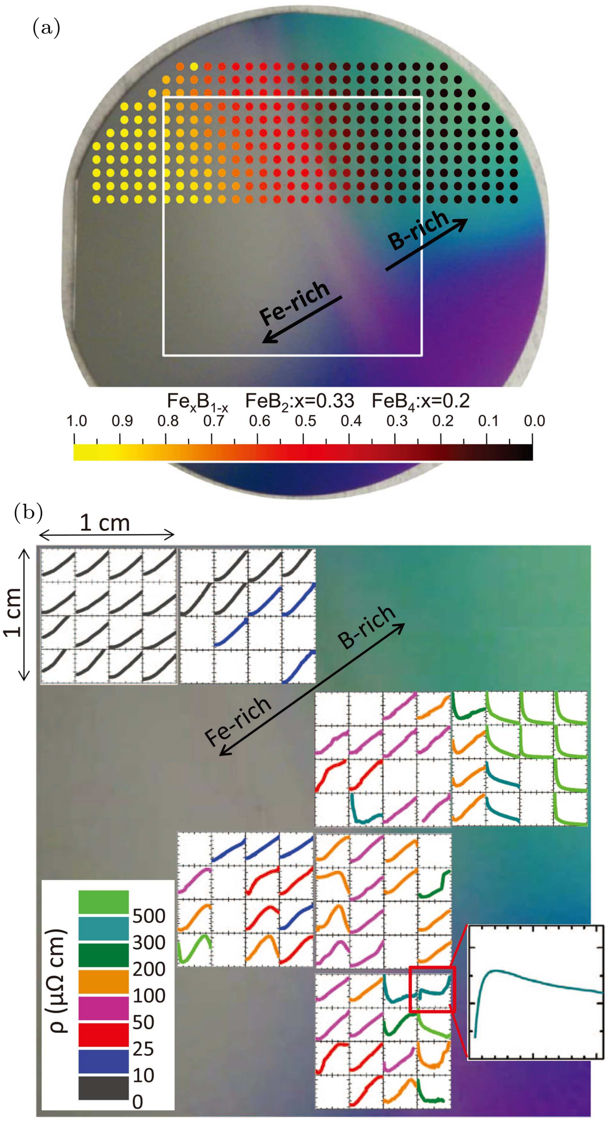

Fig. 1. (color online) Composition and resistance measurements of the Fe–B binary compositional phase space. (a) A 3-inch-diameter (1 inch = 2.54 cm) composition spread wafer. (b) Mapping of the temperature dependence of resistivity on the composition spread film.[26]

More surprisingly, one spot whose composition was approximately FeB_2 displayed a sharp resistance drop, suggesting the presence of the superconductivity under 10 K which was further confirmed by measurement of AC susceptibility and resistance in magnetic fields. In this work, the materials library following a high-throughput theoretical prediction[30] was successfully fabricated by the first generation of combinatorial thin-film preparation technology and a trace of superconductivity in the composition spread film was quickly detected. However, this kind of approach for preparing combinatorial films results in an undefined composition distribution which is disadvantageous for pinning down the useful component.

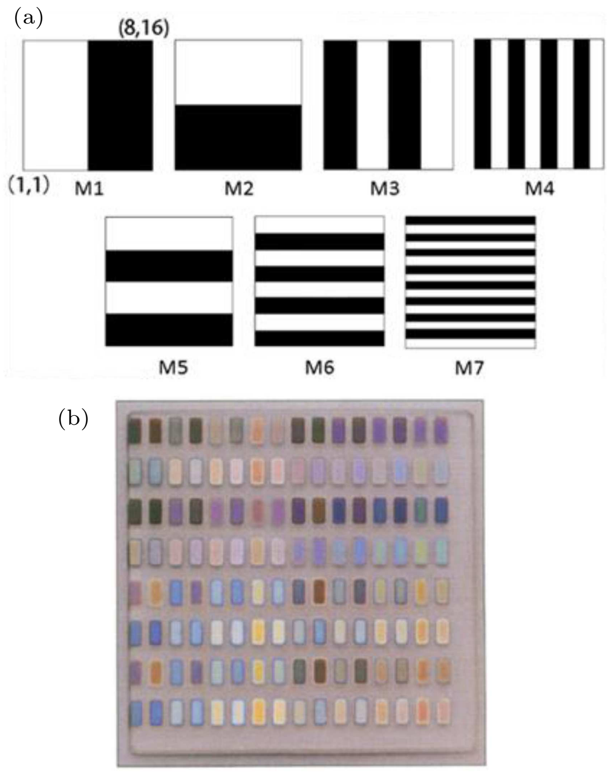

Fig. 2. (color online) Combinatorial material chip technology. (a) Binary masks used for library synthesis. The numbers at the lower left and upper right corners indicate its orientation with respect to the coordinates of the library members. (b) A 128-member binary library prior to sintering. Each site is 1 mm by 2 mm.[12]

The second generation of combinatorial films appeared in 1990s.[31] Xiang *et al.* developed a method combining thin film deposition and physical masking techniques in order to realize the parallel synthesis of spatially addressable libraries of solid-state materials. They generated material libraries on MgO and $LaAlO_3$ single-crystal substrates by sputtering target materials (CuO, Bi_2O_3, CaO, PbO, $SrCO_3$, Y_2O_3, and $BaCO_3$) through physical masks, as shown in Fig. 2(a), using a radio frequency (RF) magnetron sputtering gun. The library containing 2^n combinations can be formed by changing masks in sequence according to the precursor. A 128-member material library (see Fig. 2(b)) was prepared to further examine the effects of stoichiometry and deposition sequence on the properties of $BiSrCaCuO_x$ films. More importantly, $BiSrCaCuO_x$[32] and $YBa_2Cu_3O_x$[33,34] were found to be superconducting on one substrate, a proof of the compatibility of different families of copper oxide superconductors with a common processing condition. This pioneering work offers an improved synthetic methodology called combinatorial material chip technology for exploring superconductors. In addition, the technology has been widely applied in electronic, magnetic, optical, and dielectric materials as well as catalysts and alloys.[35] However, the fabrication process in this technique is different from an atomically controlled layer-by-layer thin-film growth which is required for precise study of material properties.[10]

Combinatorial laser molecular beam epitaxy (CLMBE), the so-called third generation of combinatorial thin-film preparation technology, was developed to carry out parallel fabrication by atomic layer-by-layer process.[10] The state-of-the-art laser molecular beam epitaxy is used in combination with the continuous moving mask technique[36] to control layering sequences which is supposed to avail the fabrication of high-quality superconducting films. The procedure for preparing binary combinatorial films with continuous chemical composition spread on a single substrate is briefly descripted as follows (see Fig. 3): When target A is ablated by laser pulses, a metal mask passes over the substrate resulting in a linear distribution of A. Target B is then ablated in order to obtain the reverse distribution by moving the mask in the opposite direction.[23] A reflection high-energy electron diffraction system for *in-situ* diagnostics of the film growth mode could also be used to ensure that the two precursors are mixed in one unit cell. Thus for mapping precise phase diagrams of materials such as sensitive copper oxide superconductors,[37] CLMBE has the obvious advantages of continuity of composition distribution, controllability of composition spread and consistency of growth condition. In 2017, Yu *et al.* fabricated the combinatorial $La_{2-x}Ce_xCuO_{4\pm\delta}$ ($x = 0.1$–0.19) thin film on an $SrTiO_3$ substrate with CLMBE.[23] They successfully obtained the doping dependence of the c-axis lattice constant and T_c by micro-region x-ray diffraction and transport measurements (see Fig. 4), respectively. It is in good agreement with the previous results from conventional uniform films.[38,39] This work clearly provides a feasible method for efficiently mapping accurate phase diagrams and exploring quantum critical points. In 2013, Wu *et al.* prepared libraries of $La_{2-x}Sr_xCuO_4$-La_2CuO_4 bilayer samples making use of combinatorial atomic layer-by-layer molecular beam epitaxy and got the relationship between chemical potential and doping,[40] which demonstrates the superiority of combinatorial films as well.

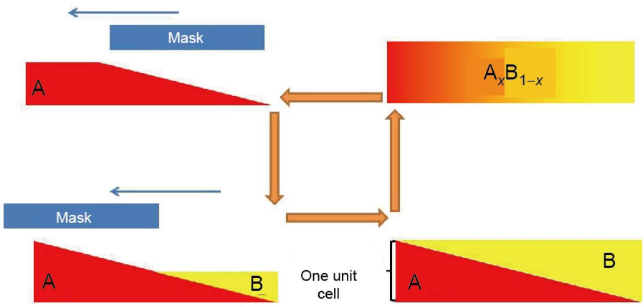

Fig. 3. (color online) Schematics of the binary-combinatorial-film growth using the continuous moving mask technique.[23]

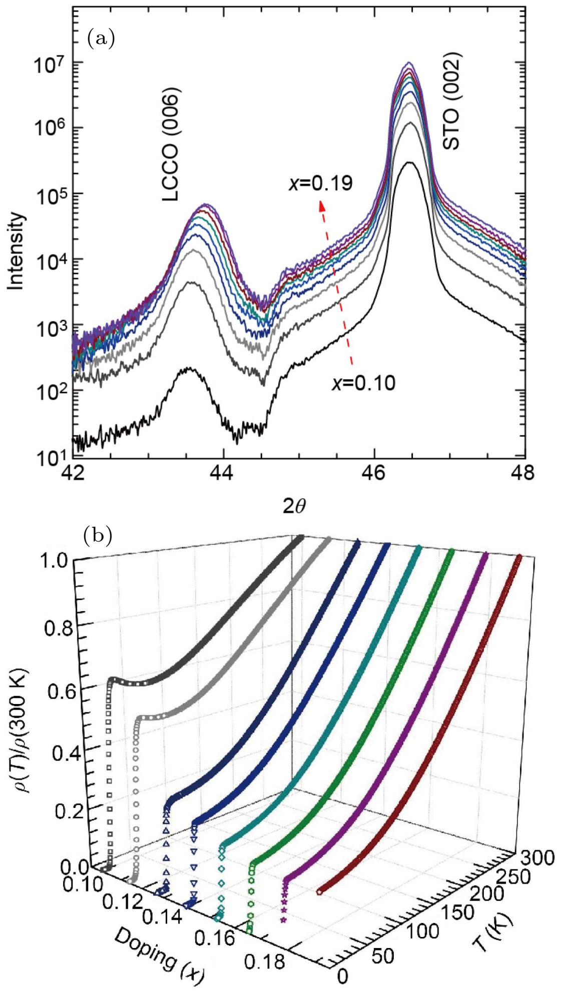

Fig. 4. (color online) The characterization of the combinatorial $La_{2-x}Ce_xCuO_{4\pm\delta}$ films. (a) Micro-region x-ray diffraction results. (b) Temperature dependence of the resistivity of different micro-regions in the film.[23]

Although the combinatorial thin-film preparation methods elaborated above provide spatially addressable arrays of compositions, the annealing process is also required for phase formation.[41] This thermodynamic process can be realized by parallel method or discrete method.[12] The parallel method implies that one piece of combinatorial film is placed at a specific temperature. It is proved to help fabricate high-quality epitaxial combinatorial materials.[42] The discrete method consists in placing points of the material library under different heating processes for the sake of high-throughput phase formation.[43] In addition, oxygen concentration plays a crucial role in cuprate high-T_c superconductors. But there is not any feasible method for high-throughput oxygen control, showing that more effort should be paid to the high-throughput preparation technology.

In addition to high-throughput preparation, rapid characterization of combinatorial films is also a key point in the research on superconductivity. At present, the tools used for high-throughput characterization always possess two basic features.[24] First, they are equipped with a highspatialresolution probe. The generalized probes can be either physical probes used in facilities like atomic force microscope and scanning tunneling microscope (STM) or beams aggregated by lenses in magnetic–optical Kerr microscope and scanning electron microscope. Second, the tools have the ability of parallel detection or high-speed scanning. In terms of probes, scanning can be realized by mechanical motion or deflection controlled by electromagnetic fields. Here, we will list several high-throughput characterization devices (see Fig. 5) expected to be extensively applied to research on superconductivity. The x-ray diffractometer (XRD) is a powerful technique for characterizing the crystal structure of materials.[44] Combined with micro-region components, high-resolution XRD can carry out quasi one-dimensional or two-dimensional scanning. Therefore, micro-region structure information of the superconducting combinatorial films can be acquired rapidly.[9,45] Ellipsometer is a kind of optical characterization method commonly used.[46,47] The imaging ellipsometer can implement visual tests of optical constants and thickness of the micro-region superconducting thin films by combining ellipsometry with conventional microscopy. In order to screen the electrical transport characteristics the parallel four-terminal method is adopted.[48,49] A multi-pin probe array is used to perform simultaneous resistance measurement at low temperature. The thermal transport signals such as Nernst and thermopower are complementary and indispensable to electrical transport in clarifying multi-band features, superconducting fluctuations and phase transitions.[50] Therefore, developing a scanning thermal transport measurement method is also of great significance for high-throughput research on superconductivity.[51] As a typical high-throughput means for the characterization of micro-region electromagnetic properties,[52,53] near-field microwave microscope has been used to measure the superconductors' microwave surface resistance,[54] directly related to pairing symmetry and quasiparticle dynamics.[55] This technology with high spatial resolution[56,57] has the potential to serve as a powerful tool for studying superconductivity.[24,58]

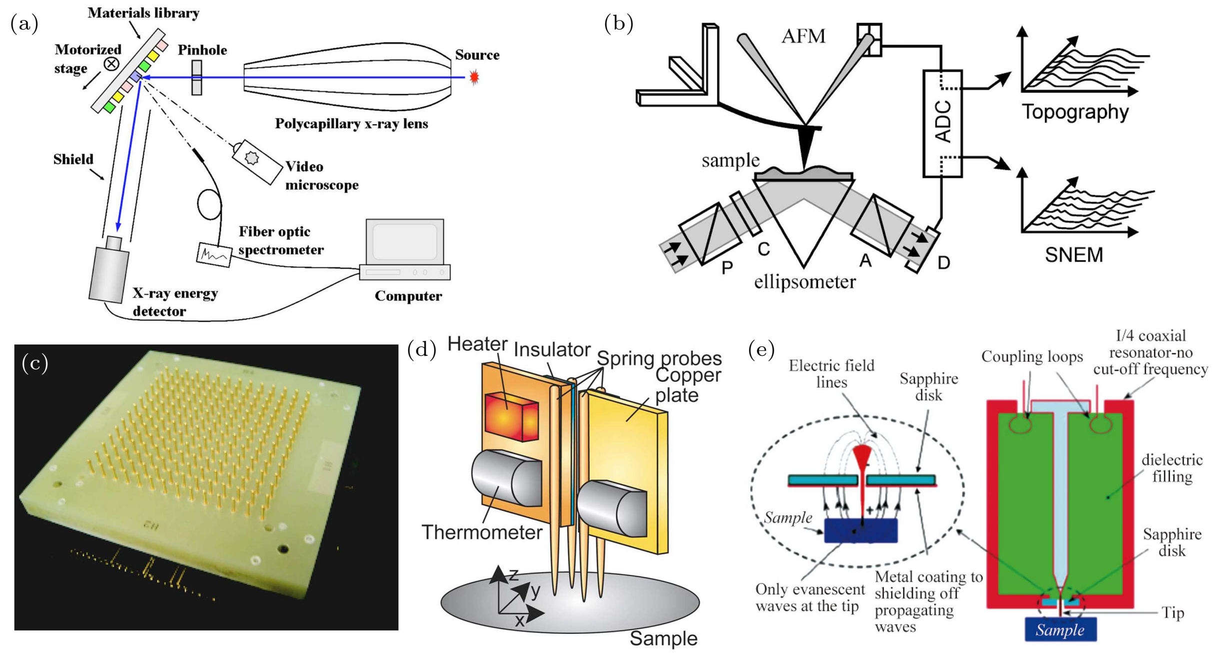

Fig. 5. (color online) Typical high-throughput characterization techniques. (a) Schematic diagram of the x-ray characterization system for combinatorial material studies.[44] (b) Experimental setup of the scanning near-field ellipsometric microscope.[59] (c) The 196-pin device for four-contact resistivity measurements.[49] (d) Schematic diagram of the probe to measure thermal transport signals.[51] (e) Schematics of the probe used in the scanning evanescent microwave microscope.[12]

3. Outlooks of high-throughput research on superconductivity

While combinatorial film preparation techniques have been developed, there is a great need to develop high-precision high-throughput characterization instruments such as scanning superconducting quantum interference device[60] and scanning magnetic force microscope.[61] Furthermore, the combined systems such as CLMBE-STM will also be beneficial to the study of superconducting combinatorial libraries because metastable thin films should be measured *in-situ*. Last but not least, the kernel of MGI is to develop new methods and research schemes which will be emphasized through a recent highlight on Fe-based superconductivity.

Since the discovery of superconductivity with $T_c \sim 26$ K in $La[O_{1-x}F_x]FeAs$,[63] there has been an upsurge of interest in studying Fe-based superconductors. Among all the Fe-based superconductors, FeSe has the simplest structure whereas its superconductivity can be tuned from $T_c \sim 8$ K in bulks[64] to ~ 77 K in monolayers.[65] Through intensive study of the remarkable enhancement of superconductivity, it was found that the superconductivity of FeSe is extremely sensitive to the ratio of Fe to Se.[66] Moreover, the variation of excimer laser fluence is likely to change the stoichiometry of films for the pulsed laser deposition (PLD) technique.[67] Based on these, Feng *et al.* successfully fabricated combinatorial FeSe films using the PLD technique.[62] They obtained a 30 mm long FeSe sample with a continuous T_c variation using two slightly sliding laser beams generating a trapezium-like distribution of beam density on the target (see Fig. 6(a)). Results of micro-region XRD (see Fig. 6(b)) and resistance measurement (see Fig. 6(c)) reveal the positive correlation between T_c and the c-axis lattice constant. These results obtained within only one week can be compared to the sketchy one obtained in more than 1000 uniform samples fabricated by conventional methods over three years.[68] Besides, it is worth mentioning that a series of high-quality uniform films with different T_c were also prepared to clarify the relation between T_c, the carrier density obtained by electrical transport measurements and the electronic structure derived from angular resolved photoelectron spectroscopy.[69]

A novel high-throughput method was developed in this work which also provided us with a brand-new high-throughput research scheme that is making use of conventional methods to carefully study the key compositions in the combinatorial material library. This notion is necessary at present because characterization tools without enough spatial resolution are not suitable for combinatorial material library and it is time-consuming to segregate the required compositions. Therefore, partly returning to conventional methods on the basis of high-throughput experiments is beneficial to the research on superconductivity. A combination of high-throughput methods advantageous in large-scale scanning and conventional techniques helpful in fine study is promising to elucidate the key ingredients of superconductivity.[70]

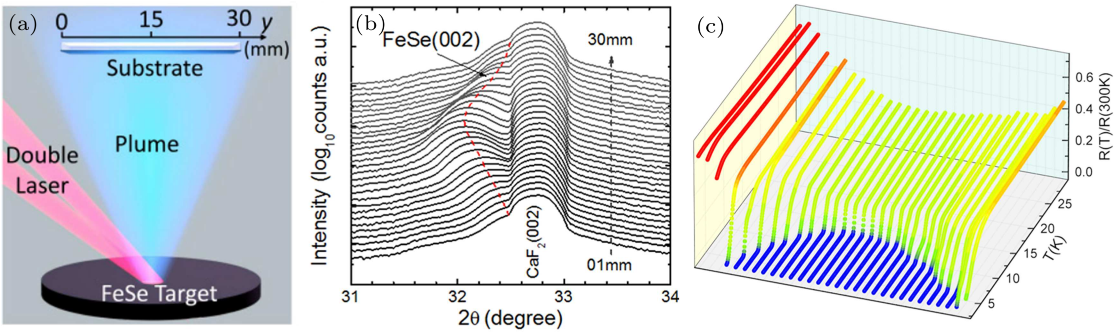

Fig. 6. (color online) High-throughput FeSe films fabrication and characterization. (a) Schematics of the double-beam pulsed laser formed via a spectroscope and focused onto the target with dislocation. (b) XRD patterns for the out-of-plane (002) peak along the *y* direction shown in panel (a). (c) Temperature dependence of the normalized resistance along *y* direction.[62]

4. Summary and prospects

The exploration of new superconductors and the study of the microscopic mechanisms of superconductivity have always been important topics in condensed matter physics. In this review, the advantages of the MGI in studying superconductors are exposed by summarizing the notion of combinatorial material sciences and some highlights about high-throughput research on superconductivity. In our opinion it is important to focus on the development of more advanced tools as well as new modes such as combining high-throughput techniques with conventional methods. There is no doubt that high-throughput equipment clusters relying on national research platforms and large-scale scientific facilities can accelerate the development of materials.

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