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Advances Research in High-Throughput MgB_2 Superconductivity

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Abstract: *Superconducting materials find applications in a rapidly growing number of technological areas, and searching for novel superconductors continues to be a major scientific task. However, the steady increase in the complexity of candidate materials presents a big challenge to the researchers in the field. In particular, conventional experimental methods are not well suited to efficiently search for candidates in compositional space exponentially growing with the number of elements; neither do they permit quick extraction of reliable multidimensional phase diagrams delineating the physical parameters that control superconductivity. New research paradigms that can boost the speed and the efficiency of superconducting materials research are urgently needed. High-throughput methods for rapid screening and optimization of materials have demonstrated their utility for accelerating research in bioinformatics and pharmaceutical industry, yet remain rare in quantum materials research. In this paper, we will briefly review the history of high-throughput research paradigm and then focus on some recent applications of this paradigm in superconductivity research. We consider the role these methods can play in all stages of materials development, including high-throughput computation, synthesis, characterization, and the emerging field of machine learning for materials. The high-throughput paradigm will undoubtedly become an indispensable tool of superconductivity research in the near future.*

Keywords: Superconductivity, High Throughput, Machine Learning, Combinatorial Film, Rapid Screening

I. INTRODUCTION

Ever since Kammerlingh-Onnes in Leiden observed the disappearance of electrical resistance of mercury at very low temperatures in 1911, the phenomenon of superconductivity has been intensely studied. Yet, the exact nature of this effect remained unclear until the Bardeen-Cooper-Schrieffer (BCS) theory was developed in 1957 [1, 2]. Establishing the physical parameters behind the critical temperature (*T*c) then became one of the main research topics in the field. Observation of an isotope effect clearly demonstrated that *T*c in most then- known-superconductors is linked to the coupling between the electrons and the lattice, with the strength of the electron- phonon (el-ph) interaction playing a key role. Increasing el-ph coupling enhances *T*c as long as the atoms in the crystal only vibrate near their equilibrium sites. But as the coupling strength crosses from a weak regime to an intermediate regime, the normal metallic state itself becomes unstable and a competing state such as a charge density wave may arise, causing structural changes. Although early analyses pointing to a ceiling of *T*c at about 30 ~ 40 K proved too simplistic, it quickly become clear that achieving significantly higher critical temperatures would require unusual conditions or materials [3]. (One example is the recent remarkable observation of high *T*c in hydrogen-rich materials under extreme pressure [4]

In 1986, the ceramic compound Ba-La-Cu-O was found to become superconducting at 35 K by Bednorz and Müller in Zurich [5]. This milestone work not only brought hope for superconductors with *T*c above 40 K, but also inspired other physicists to expand their search by exploring compounds with complex crystal structures and containing multiple elements. Soon, the limit of 40 K was crossed [6, 7]; critical temperatures above that of liquid nitrogen (77 K) were realized [8, 9], and the highest *T*c reached 138 K in Hg0.8Tl0.2Ba2Ca2Cu3O8.33 (at ambient pressure) [10]. While these discoveries inspired a generation of researchers, the mechanism of superconductivity in this class of materials (dubbed cuprates) is still a mystery.

Before the discovery of high-*T*c cuprate (HTSCs) materials, most known superconductors were composed of no more than two elements and were predominantly elemental superconductors, alloys, and intermetallics. As the superconducting transition temperature went beyond the 40 K mark, the superconducting compounds became more

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complex and containing more elements; for example, YBa2Cu3O6+δ, HgBa2Ca2Cu3O8+δ and Hg0.8Tl0.2Ba2Ca2Cu3O8.33 are made up of 4, 5 and 6 elements respectively. On the other hand, the recent discoveries of Fe-based [11, 12], Cr-based and Mn- based superconductors [13-15] demonstrated that even compounds with elements once thought strongly detrimental to superconductivity have to be considered. Obviously, the number of possible combinations grows exponentially as more and more elements are added in the candidate materials. Thenumber of compounds can be easily estimated within the group of natural elements; the order of magnitudes for possible combinations are 10^3, 10^5, 10^7 and 10^9 for binary, ternary, quaternary, and penternary compounds respectively. In addition, many other methods to modulate superconductivity are known: ultra-high pressure [16, 17], ultra-thin film deposition [18], superlattice architecture [19] and ionic liquid/solid gating [20, 21] are a few examples. Third, some HTSC materials are extremely sensitive to physical and chemical parameters. For instance, one percent variation in cation content can turn a copper oxide superconductor into an insulator [22]. One will have to do a vast amount of synthesis to construct a reliable and detailed phase diagram as a function of cation and anion contents [23]. What is more, a complete phase diagram of HTSC must necessarily be multivariate, i.e. alongside cation and anion substitutions it must include synthesis conditions, pressure, magnetic field, and other variables. It is clearly not feasible to efficiently construct such a multidimensional phase diagram using the traditional one-material-at-a-time experimental methodology.

In general, there are two main challenges in the experimental study of superconductivity: i) searching for novel superconductors in the enormous space of candidate compounds comprised of more and more elements; ii) delineating the key physical parameters that control the superconductivity, by means of establishing a reliable multidimensional phase diagram. Conventional experimental methods are not well suited to address these problems, and new paradigms and tools are required to boost the efficiency of superconductivity research [24, 25].

In bioinformatics and the pharmaceutical industry, similar challenges arose before the discovery of HTSCs. There is a huge number of possible gene combinations and drug formulas, and it is not realistic to synthesize and test them one by one. The idea of doing many tests at the same time – the so-called high-throughput strategy – was rapidly developed and put into practice. Ever since, high-throughput methods have been a key driving force behind the development of modern biology and medicine. This methodology was also gradually adopted in condensed matter physics and materials science.

The general high-throughput materials research procedure has the following steps - sample synthesis, characterization, analysis of the data. Significant increase of the efficiency necessitates the acceleration of every step, as well as substantial coordination among them. The high-throughput approach for

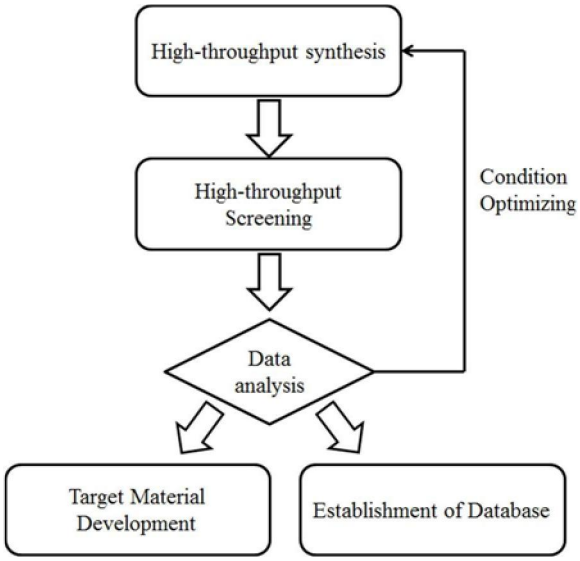

Figure 1. The high-throughput approach for discovery and development of materials.

In 1970, Hanak introduced the rudiments of a multiple- sample-concept in materials research, by developing a multiple-target, radio-frequency co-sputtering deposition [26]. However, the difficulties in obtaining and processing a large

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amount of experimental data prevented the wide-spread adoption of high-throughput methods in materials research at the time. Another factor inhibiting the spread of these methods was the low demand for complex functional materials. This has dramatically changed in the last decades, owing to the needs of industries such as communications, aerospace, energy, and transportations. The demand for novel materials has experienced an explosive growth, both in terms of diversity and required performance. This necessitated significant acceleration of materials research and development. High-throughput synthesis and characterization technology became a major research tool, leading to a substantial progress in a number of applications (see, for example, Refs. [27-34]). Helping these advances, the rapid progress in information technologies created powerful tools able to deal with the massive amounts of data generated by this approach.

Thus, high-throughput paradigm provides an extremely promising approach to meet the challenges in superconductivity research outlined above. In one early pioneering work demonstrating the potential of these methods, Xiang *et al.* reported the rapid synthesis of high-*T*c superconductors, with a 128-member library of copper oxide superconducting thin films deposited on a single substrate in one batch [35]. Although the superconductors synthesized in this work had been known already, the increase in efficiency achieved by the parallel synthesis was encouraging. In 2013, Jin *et al.* provided the first example of a superconductor discovered via the high-throughput methodology. They fabricated composition-spread films comprised of Fe and B, across 3-inch Si wafers by a co-sputtering technique.Indications of a superconducting region were found from the resistance measurement by a home-made probe with an array of 64 pogo-pins (figure 2b). Superconductivity was then confirmed by a zoom-in magnetotransport and susceptibility measurements [36]. In 2016, Wu *et al.* studied a library of heterostructures made up of La2CuO4 and combinatorial La2- *x*Sr*x*CuO4 ($0.15 \leq x \leq 0.47$), achieved by varying the deposition rate of the Sr content in an advanced oxide molecular beam epitaxy system [37]. In 2018, Stanev *et al.* filtered more than 100,000 compounds and created a list of potential superconductors, using a machine learning models trained on the critical temperatures of more than 12,000 known superconductors [38].

As seen from these examples, the high-throughput paradigm is already starting to permeate superconductivity research. In this review, we outline the advances in all parts of high-throughput superconductivity research: high-throughput synthesis (Section 2), high-throughput characterizations (Section 3), high-throughput *ab-initio* calculations (Section 4), and machine learning (Section 5). We also discuss the need to develop a next generation of facilities, techniques, and platforms in order to fully utilize the power of high-throughput methods (discussed in Section 6) [36].

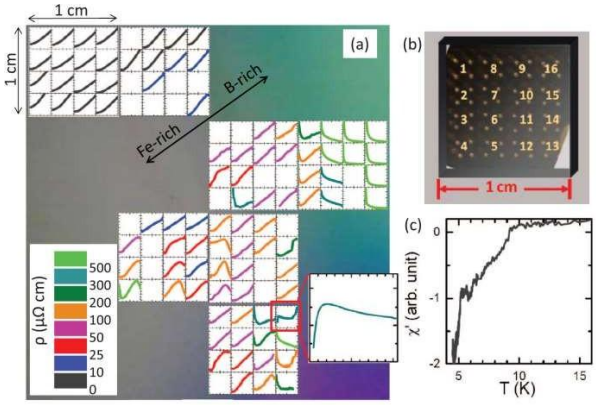

Figure 2. Mapping of the temperature dependence of resistivity on the Fe-B composition spread film.

II. HIGH THROUGHPUT SYNTHESES FOR SUPERCONDUCTORS

High-throughput synthesis is one of the foundations of the recent acceleration of the rate of materials exploration. However, that does not mean the synthesis cost has suddenly become irrelevant; efficiency is a key point – the speed of synthesis should grow faster than the resources it demands. To achieve this, common components and procedures used in different experimental steps, like evacuation, heating and conditioning atmosphere, should be combined as much as possible. This is the basis of the so-called combinatorial approach, which is the most efficient high-throughput synthesis method. Koinuma *et al.* gave the basic concept of combinatorial chemistry for solid state materials in Ref. [39]. During synthesis, a chemical reaction for the target material is influenced by many factors. The search for new materials can be

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regarded as a process of scanning certain points in the phase diagram as a function of multiple variables, as shown in figure Compared to the classical synthesis process of an effective point-by-point search, combinatorial approach can much faster map key parameters (such as composition, temperature, and pressure) responsible for optimizing a functionality.

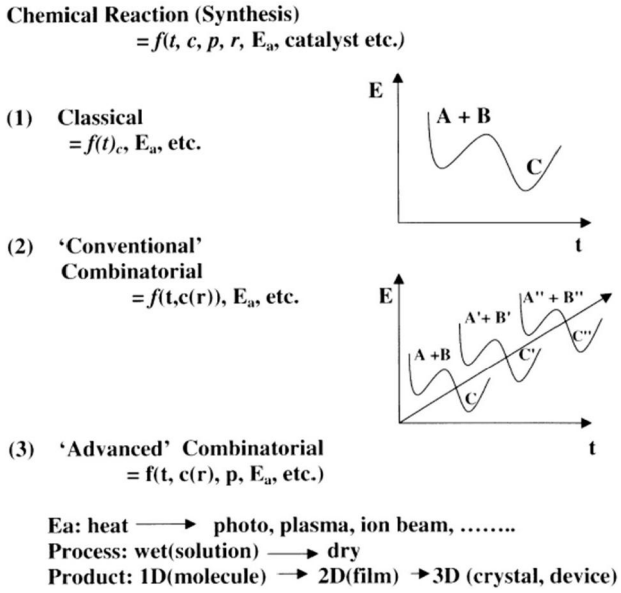

Figure 3. Concepts of combinatorial synthesis versus classical synthesis [39].

Thin-film preparation technology is the most widely used combinatorial synthesis method [30]. As early as 1965, Kennedy *et al*. reported a method for rapidly obtaining a Fe- Cr-Ni ternary library by electron beam co-evaporation of three precursors (Fe, Cr and Ni targets), on an equilateral-triangle metal foil with a side length of 10 inches [40]. Three generations of combinatorial film growth techniques have been developed since then. The first generation realizes a gradient chemical composition by ablating the precursors simultaneously and mixing them using natural diffusion. Both co-evaporation [40, 41], co-sputtering [26, 42, 43] and co- laser ablating [44] techniques are categorized into this generation. Figure 4 shows a typical configuration of a co- sputtering system. Different sources are mounted against the wafer at a certain angle to the normal direction of the wafer surface. During the deposition, such configuration results in a spatial variation of the deposition rate and allows plumes from different targets to overlap and form a natural composition gradient. Recently, Jin *et al.* obtained an Fe-B binary spread by such method, with a continuous composition across a 3- inch Si wafer with a 200 nm SiO2 layer on top [36]. The lower part of figure 4 shows the photograph of one compositionspread wafer taken under natural light; the average composition at different positions (solid circles) is obtained by the wavelength dispersive spectroscopy (WDS). A key advantage of the co-deposition method is that it requires relatively simple facilities and the deposition process is easy to control. However, the chemical composition cannot be precisely controlled because of the natural variability of the spatial deposition rate. The local composition of the film can only be determined by micro-region analysis methods (discussed in the section of high-throughput characterizations).

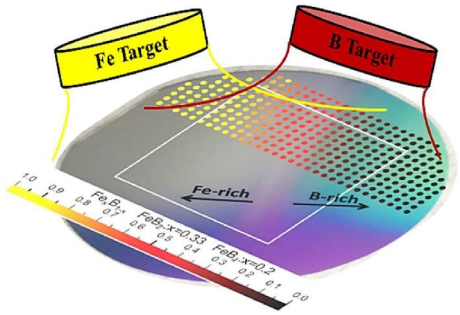

Figure 4. Sketch of the co-sputtering deposition for an Fe-B composition spread combi-film.

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Second-generation methods were developed to overcome the shortcomings of the co-deposition approach. Xiang *et al.* used a method combining thin film deposition and physical mask techniques for a parallel synthesis of spatially addressable libraries of materials [35]. The programmable mask is at the core of the second-generation technology. Films fabricated with these methods contain discrete squares with varying compositions. Because these small regions are addressable, such films are also called integrated materials chips or combinatorial materials libraries. More details about this synthesis method can be found in the earlier literature (e.g. [32, 45-47]). It has to be noted that the mask is not necessarily a physical one. Wang *et al.* developed a combinatorial robot which is able to create thick-film libraries by ink-jet printing, as shown in figure 5 [48]. After being ground into sufficiently fine powder, precursors can be made into inks [49], taking the role of the three "primary colors". Different doses of inks are ejected on designated places of the wafer. Thus, using selected precursors and an appropriate post processing, an addressable materials library can be created. In this way, the ejector together with the robot arm replaces the physical mask [50, 51].

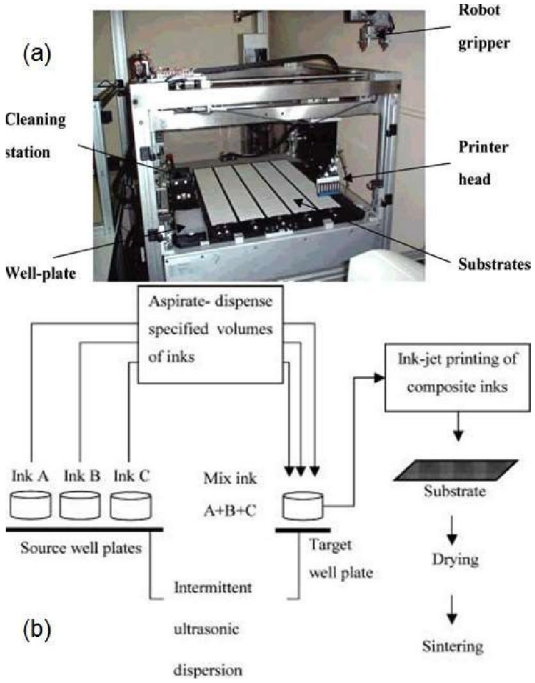

Figure 5. (a) The ink-jet printer table within the robot gantry, populated with alumina substrates. The pick and place robot arm is seen in the top right. (b) Schematic diagram of the mixing and printing protocol [48].

Mask patterns has been widely utilized in the study of electronic, magnetic, optical and dielectric materials, as well as catalysts and alloys [52]. However, for more precise studies of material properties, distinct techniques such as atomically controlled layer-by-layer thin-film growth are required [25].

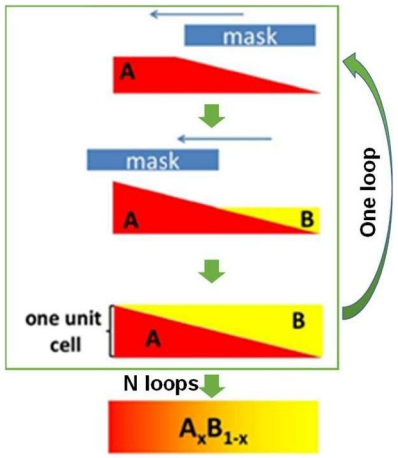

Figure 6. Schematic of binary-combinatorial-film growth using the continuous moving mask technique [53].

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Combinatorial laser molecular beam epitaxy (CLMBE)–the so-called third generation of combinatorial thin-film preparation technology–was developed to carry out a parallel fabrication via a layer-by-layer growth mode [25]. The state-of-the-art laser molecular beam epitaxy in combination with a mobile mask technique has been already used to control layering sequences of high-quality superconducting films [54]. The procedure for preparing binary combinatorial films with continuous chemical composition spread on single substrate can be briefly described as follows (see also figure 6): once a target A is ablated by laser pulses, a metal mask is moving along the substrate in half a period time and results in a linear distribution of the A component. In the other half period, the other target B is ablated for a reverse distribution by moving the mask in the opposite direction [53]. A desired thickness of the combinatorial film can be achieved by setting corresponding periods of deposition. It should be emphasized that the two precursors A and B must be mixed in a single unit cell in the period, monitored by the reflection high-energy electron diffraction system for *in-situ* diagnostics. Otherwise, a superlattice rather than a combinatorial film will be obtained.

CLMBE is easy to operate, and benefits from a wide chemical composition range in one batch of deposition. This makes it perfect for obtaining a precise phase diagrams of materials like cuprate superconductors [5], For instance, Yu *et al*. fabricated combinatorial La*2-x*Ce*x*CuO4±δ thin film with *x* from 0.1 to 0.19 on 1×1 cm^2 SrTiO3 substrate by the CLMBE [53].

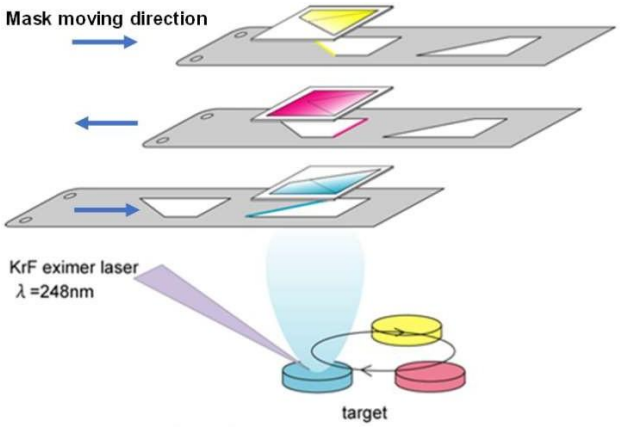

Figure 7. (From the introduction brochure of Pascal Co., Ltd in Japan) Schematic of ternary-combinatorial film growth using the continuous moving mask technique. The blue arrows indicate the direction of mask motion in every step.

Following the same principle, a ternary-combinatorial film can be grown with ablating alternatingly three targets and shadowing the substrate from three directions by mask with a sophisticated pattern design (shown in figure 7). With this method, Mao *et al*. prepared an Mg–Ni–Al ternary thin film library [24]. In combinatorial films, the chemical composition is locked to the fixed space point of the sample surface. For a ternary compound, the formula can be written as A*x*B*y*C1-*x*-*y*, in which A, B and C stand for different elements. Since there are two independent spatial coordinators (e.g. *x* and *y*)

determining the position corresponding to a unique chemical composition, the full ternary library can be obtained based on single combinatorial spread. However, if more than two variables are involved, e.g. three variables in the quaternary A*x*B*y*CzD1-*x*-*y*-*z*, one cannot obtain all the combinations of these four components in a single chip; instead, a pseudo multi- component materials library can be created.

As explained above, three generations of distinct combinatorial-film preparation techniques have been developed. It has to be noted that each generation has its own unique merits, and is suitable for different kinds of materials. Sometime, a customized combinatorial approach is required, for example for compounds that only show superconductivity in a very narrow range of chemical doping. A typical iron- based superconductor, FeSe1-*δ*only displays superconductivity within a finite *δ* [55]. Since Se is a volatile element, fine control of the Fe/Se ratio of the final film can be extremely challenging by the methods presented above. Recently, a double-beam laser configuration was used to produce a redistributed laser flux density on a FeSe target. As reported in the study of SrTiO3 films grown by pulsed laser deposition (PLD), the stoichiometric ratio can be regulated by the power density of the laser [56]. Tuning the parameter of the combinatorial laser, the Fe/Se ratio of the final film can be varied by a very narrow δ step (i.e. the order of magnitude is ~ 0.001, which is beyond the resolution limit of the common chemical analysis methods). The left panel of

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figure 8 shows the schematic map of double-beam laser used in the deposition; the corresponding evolution of the superconducting critical temperature can be clearly seen in the normalized *R-T* curves from different space regions of the final film (right panel). Moving along the film from one edge to the other, *T*c changes continuously from below 2 K to 12 K and back to below 2 K. There is no obvious difference in the film thickness (~150 nm) across the whole film (verified by scanning electron microscope (SEM)); thus, thickness influence on *T*c can be excluded. Such combinatorial films offer a good platform to investigate the nature of tunable superconductivity in the Fe- Se binary system [57].

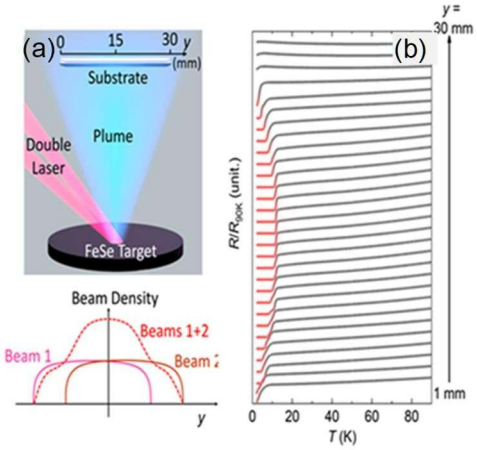

Figure 8. (a) Schematic process of the double-beam pulsed laser deposition via spectroscope, and then focused onto the target with controllable displacement. (b) The temperature dependence of normalized resistance along the ***y*** direction, with the highest *T*c in the middle [57].

Wu *et al.* used a tilted Knudsen cell in an oxide molecular beam epitaxy system to synthesis combinatorial La2-xSrxCuO4 film. Although in principle this falls into the class of first generation combinatorial techniques, it provides a much finer control over the composition. The deposition rate can be precisely tuned and the growth process can be monitored in- situ. The composition gradient originates in the angle between the sources and the substrate, as shown in figure 9. By means of this method, the composition across the film can be confined to extremely fine steps, Δx ~0.00008 [37, 58].

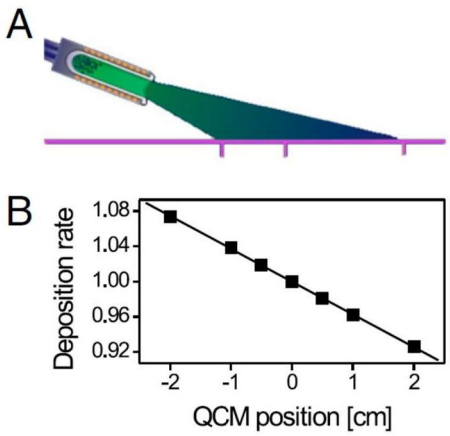

Figure 9. Schematics of the deposition geometry: thermal effusion cell is positioned at a shallow angle (20°) with respect to the substrate. Closer to the source, the deposition rate is higher [37].

In addition to generating composition-spread material libraries, combinatorial approaches can also be used to rapidly ascertain the optimal growth condition for superconducting materials. For example, the conventional way to find the best deposition temperature of a superconducting material is to try different temperatures one by one. However, a much more efficient method is to create a thermal gradient on the substrate during film deposition [59]. Figure 10 shows one configuration which can create such gradient during the growth process. One end of the substrate is attached to a heater while the other end is kept free-standing. When the heater is set at high temperature, heat diffuses to the free-standing end through the substrate. Using such configuration, a temperature range in which only pure β-FeSe phase forms was

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found; only three batches of samples were tested with deposition temperature from 350 to 700℃ [60].

In addition, parallel synthesis can help reduce the uncertainty originating in the difference of growth conditions, unavoidable between separate experimental batches. Figure 11 shows the relationship between the out-of-plane crystal lattice parameter (*c*-axis) and *T*c extracted from more than one thousand pieces of uniform FeSe films (upper panel) as well as one combinatorial film (lower panel), respectively. It took almost three years to prepare and characterize the 1000+ uniform films; this is in contrast with the several weeks needed

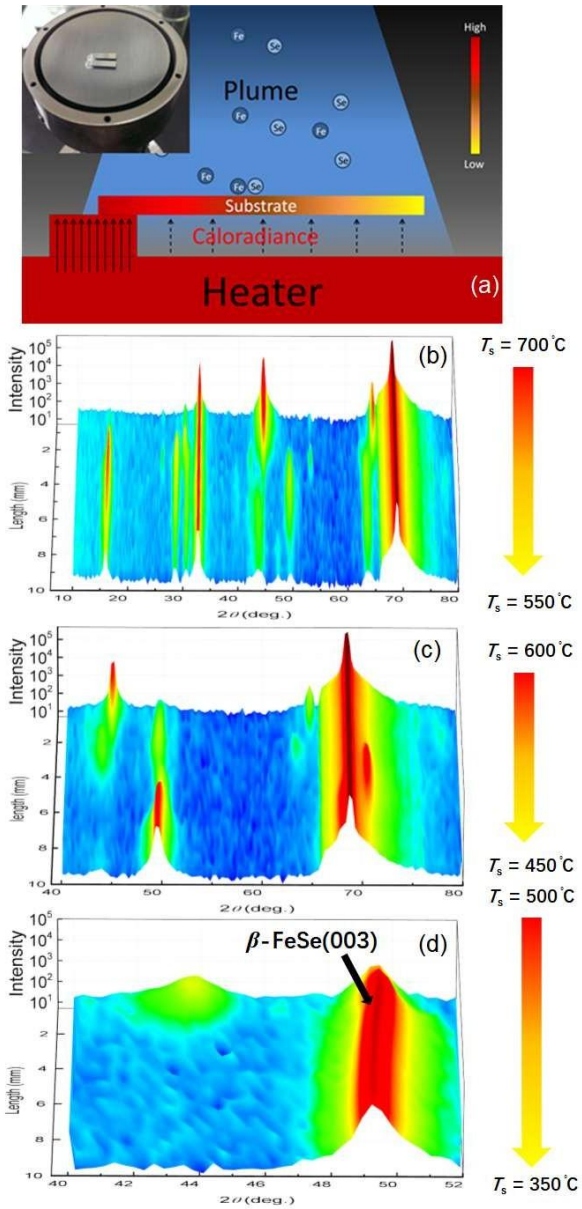

Figure 10. (a) Schematic of the combinatorial film growth with gradient temperature, inset on top left is real photo of the substrate mounted on the heater. (b-d) the micro-region *θ/2θ* x-ray patterns of the FeSe films grown using different temperature gradients:700 □-550 □,600 □-450 □ and 500 □ to 350 □, respectively. Pure *β*-FeSe phase can be found in the region where the deposition temperature is not beyond 450 □ [60].

In addition, parallel synthesis can help reduce the uncertainty originating in the difference of growth conditions, unavoidable between separate experimental batches. Figure 11 shows the relationship between the out-of-plane crystal lattice parameter (*c*-axis) and *T*c extracted from more than one thousand pieces of uniform FeSe films (upper panel) as well as one combinatorial film (lower panel), respectively. It took almost three years to prepare and characterize the 1000+ uniform films; this is in contrast with the several weeks needed for the single combinatorial film. As can be seen,

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the amount of data collected from the uniform films is much larger, however, the positive relation between *c* and *T*c0 is manifested much clearly in the combinatorial film data.

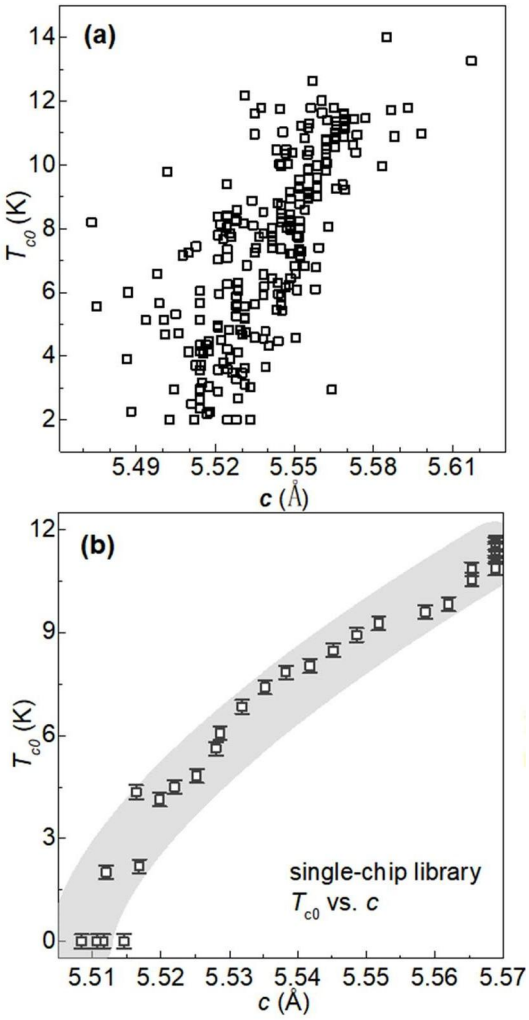

Figure 11. The *c*-axis lattice constants dependence of *T*c0 for （a) uniform FeSe films, （b) combinatorial FeSe film with gradient *T*c [57].

Combinatorial film data provides a better correlation between *T*c and lattice parameters due to its more precise composition control.

III. HIGH THROUGHPUT CHARACTERIZATIONS OF SUPERCONDUCTORS

As another step in the high throughput methodology, rapid characterization of combinatorial films is indispensable in the drive to accelerate superconductivity research. Because to the nature of combinatorial synthesis, characterization have to be conducted by probes with spatial resolution capabilities. Several commercially available probes satisfy this requirement: atomic force microscope, scanning tunneling microscope (STM), optical microscope, scanning electron microscope are just a few examples. These tools with auto scan functionality can have high spatial resolution, and are equipped with tips or aggregated beams. There are also diverse homemade probes designed to study various properties of high-throughput samples. In this section, we focus on techniques relevant to superconductivity research, briefly summarized in the following order.

3.1 Composition and Structure

Common probes used for composition and structure analyses are: x-ray diffraction; scanning electron microscopy (SEM); transmission electron microscopy (TEM). Because the electron beam can be focused, methods using it naturally

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provide the required spatial resolution and can be used for high-throughput characterizations [33, 61].

Great efforts have been dedicated to developing spatially resolved x-ray-based characterization methods. Since x-rays have much higher frequency and photon energy than visible light, they tend to penetrate or get absorbed in most materials. Unfortunately, most of the techniques used to redirect x-rays are unable to produce well focused beams.

Initially, pinholes or slits made of anti-radiation materials were used to shrink the profile size of x-ray beams. Figure 12 shows the data collected from a combinatorial La2-xCexCuO4 library chip (an electron-doped copper oxide superconductor, x varies from 0.10 to 0.19). Micro-area scan is realized by adding a narrow slot and a moving sample stage to a commercially-available x-ray diffractometer [53]. The calculated c-axis lattice constant monotonically decreases with raising the nominal doping level x, in accordance with the tendency extracted from uniform LCCO thin films fabricated by conventional pulsed laser deposition (PLD) method [62, 63].

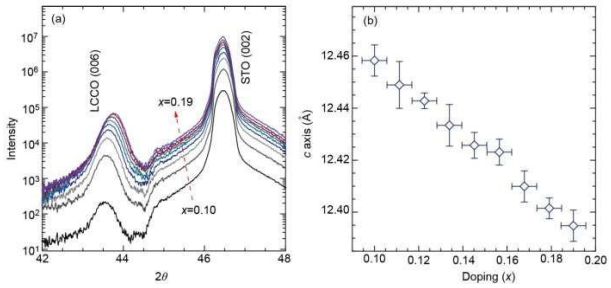

Figure 12. (a) The micro-region x-ray diffraction results of combi- film La2-xCexCuO4±δ. The component interval Δx is about 0.012. (b) The variation of c-axis lattice constant with increasing doping levels. In this figure, x refers to nominal doping level [53].

In this technique the analysis sensitivity and accuracy are unavoidably low, due to the limited incident x-ray throughput,New high-throughput techniques. While many combinatorial film preparation techniques have been developed, there is a great demand for high- precision characterization instruments, especially ones that can perform advanced spectroscopy measurements such as STM and angle resolved photoemission spectroscopy (ARPES). This requires combinatorial material libraries in the form of films to be measured *in-situ*. Accordingly, combined systems such as combinatorial-film-STM and combinatorial- film-ARPES tools will be optimal. These will be especially beneficial for the study of superconducting combinatorial libraries. Characterization methods taking advantage of synchrotron sources should become widespread; these techniques allow the study of not only composition and structure of materials, but also their elementary excitations. For instance, resonant inelastic x-ray scattering (RIXS) technique can be used to measure spin excitation [150], charge excitation [151], phase of the order parameter [152], etc. Importantly, thin films can be probed with RIXS, since it only needs small sample volumes. Compared with the neutron scattering techniques, RIXS appears much more promising in high-throughput research on superconductivity[153].

IV. CONCUSION

However, in order to maximize the efficiency and impact of high-throughput methodology, it is necessary to closely coordinate the theoretical and experimental stages and combine computations with experiments and analysis of materials databases . One possible way of doing this is to integrate all the facilities in a big platform and organize them as nodes in an assembly line. The collected data can be standardized to establish a sharable databank. Techniques such as STM, neutron scattering and ARPES can contribute to the data collection according to their limitations and characteristics. Since the MGI project was first announced in the US in 2011 [155], several similar programs have been initiated in different countries. Accelerated Metallurgy (AccMet) project initiated by the European Union and “MatNavi” database managed by National Institute for Materials Science in Japan are programs established to develop novel materials research and developments procedures. Among these programs, the Center for Materials Genome Initiative (CMGI) in Beijing, China may be the only one currently organized as a specialized high-throughput material research platform. This platform contains three large substations shown in figure 28: high-throughput computation and database station, high-throughput synthesis and fast characterization station, and high-throughput technique research and development station. MGI and similar platforms have the potentials to revolutionize materials research in the near future.

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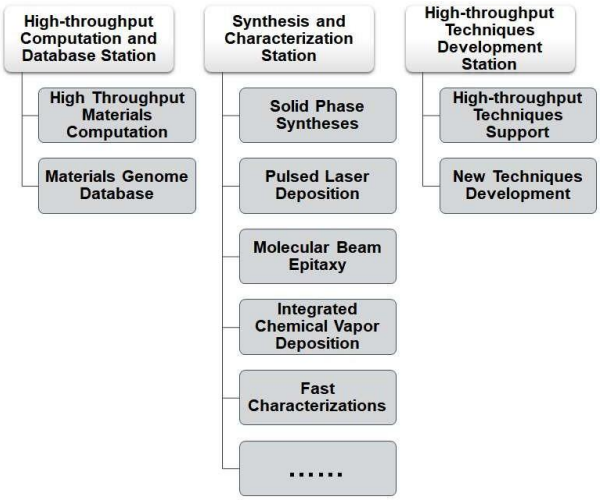

Figure. The organization of Center for Materials Genome Initiative, Huairou, Beijing.

In conclusion, integrating a novel research paradigm with new techniques, facilities, and platforms, high-throughput methodology is becoming more and more prominent in advancing the study of superconducting materials.

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