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Combinatorial approaches for high-throughput characterization of mechanical properties

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ABSTRACT

Since the first successful story was reported in the middle of 1990s, combinatorial materials science has attracted more and more attentions in the materials community. In the past two decades, a great amount of effort has been made to develop combinatorial high-throughput approaches for materials research. However, few high-throughput mechanical characterization methods and tools were reported. To date, a number of micro-scale mechanical characterization tools have been developed, which provided a basis for combinatorial high-throughput mechanical characterization. Many existing micro-mechanical testing apparatuses can be pertinently modified for high-throughput characterization. For example, automated scanning nanoindentation is used for measuring the hardness and elastic modulus of diffusion multiple alloy samples, and cantilever beam arrays are used to parallelly characterize the thermal mechanical behavior of thin films with wide composition gradients. The interpretation of micro-mechanical testing data from thin films and micro-scale samples is most critical and challenging, as the mechanical properties of their bulk counterparts cannot be intuitively extrapolated due to the well-known size and microstructure dependence. Nevertheless, high-throughput mechanical characterization data from combinatorial micro-scale samples still reflect the dependence trend of the mechanical properties on compositions and microstructure, which facilitates the understanding of intrinsic materials behavior and the fast screening of bulk mechanical properties. After the promising compositions and microstructure are pinned down, bulk samples can be prepared to measure the accurate properties and verify the combinatorial high-throughput characterization results. By developing combinatorial high-throughput mechanical characterization methods and tools, in combination with high-throughput synthesis, the structural materials research would be promoted by accelerating the discovery, development, and deployment of high performance structural materials, and by providing full spectrum of materials data for mapping composition-microstructure-mechanical properties. The latter would significantly improve the advanced structural materials design using materials genome engineering approach in the future.

Contents

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1. Introduction

Materials research and development has always been focused on establishing the composition-processing-structure-properties relationship, which so far relies mainly on the time-consuming trial-and-error experimental process. The discovery-to-deployment cycle of typical materials may range from 10 to 20 years, which has increasingly become a major bottleneck for modern technology development. In order to facilitate the materials R&D, there have been a number of innovative efforts on both high-throughput materials experimentation and computational materials designs over the past two decades.

In the middle of 1990s, Xiang et al. at Lawrence Berkeley National Laboratory reported a high-throughput combinatorial experimental approach to screen novel thin film materials [1]. In this approach, a large number of samples with different compositions were synthesized in a matrix form on a single substrate and characterized simultaneously or sequentially by imaging or fast scanning. The data were processed by computer and reported in graphic or tabular form for quick and convenient materials screening. In this integrated approach, a materials library was quickly fabricated and investigated on a single chip in a systematic, fast, and cost-efficient manner, which greatly increases the throughput and efficiency of materials discovery. Since Xiang's work was published as a front cover article on *Science*, combinatorial high-throughput experimental methods have received enormous attention in the materials community. A number of high-throughput experimental tools were successfully developed and applied to accelerate the materials R&D in many industrial fields [2–7]. Furthermore, combinatorial methods were also proved instrumental in fundamental materials science. For example, a ternary phase diagram covering the entire compositional space can be easily constructed by depositing continuously composition-graded thin film samples onto a single substrate [8–10]. This type of phase diagram consists of rich information of the composition–structure–property of novel complex materials, which are highly effective in understanding the science of new materials systems.

Advanced structural materials such as high temperature and high strength alloys often have very complex composition, sometimes consisting up to tens of elements, and structure, which make them particularly suitable for high-throughput combinatorial experimentation. However, there are not many combinatorial high-throughput experimental methodology and tools developed for structural materials in the past [11–13], partly due to the difficulty in fabricating combinatorial samples as well as measuring their mechanical properties quickly and accurately.

Three issues need to be addressed in order to achieve accurate and high-throughput mechanical characterization. Firstly, high-throughput characterization often requires combinatorial sample arrays or continuously composition-graded samples integrated in a relatively small area. The geometry of individual samples is therefore critical, which has to be small enough to be integrated and large enough to represent the characteristic properties of the targeted materials. Driven by the microelectronics and micro-electro-mechanical system (MEMS) industry, the mechanical properties of thin films and nanomaterials have been studied intensively over the past decades [14]. There are a number of techniques developed to test the mechanical properties of small scale materials and structures, e.g., nanoindentation, micro-tensile and compression test, bulge measurement, and wafer curvature measurement. Some of the techniques have demonstrated the capabilities of fabricating and testing combinatorial sample arrays on a single substrate. For example, nanoindentation is a widely used technique for hardness and elastic modulus measurement of micro-scale materials, which is suitable for both sample arrays and continuously composition-graded samples [12].

Secondly, high-throughput mechanical characterization requires fast testing of combinatorial samples in either parallel mode or scanning mode. Tools shall be highly automated for fast data acquisition and processing. Recently, Vlassak et al. developed an array of MEMS-based micro cantilever beams to study the mechanical properties of thin films in a simultaneous and parallel mode [13]. Besides, nanoindentation is a well-known scanning testing technique to measure mechanical and tribological properties of thin film materials [12]. With proper modification, other existing micro-mechanical testing tools can also reach high speed characterization for combinatorial samples.

Thirdly, data interpretation for combinatorial samples needs to be very careful to ensure accuracy and reliability. For example, size effect is well-known complication when characterizing the mechanical properties of small scale materials [15–17]. The typical small dimension of individual samples in the combinatorial materials library may cause their mechanical behavior to be very different from that of their bulk counterparts. Even though theoretical and experimental understanding of size effects has advanced significantly, accurately correlating the results from micro-mechanical testing and the actual performance of structural materials in real world applications still remains a challenge.

This paper reviews typical micro-mechanical testing techniques and analyzes the feasibility of modifying these techniques for high-throughput characterization of mechanical properties. In particular, a data interpretation strategy is proposed for reliable prediction of the mechanical behavior of bulk materials by taking the size effects into proper account.

2. Typical micro-mechanical testing techniques

Conventional mechanical test for bulk materials usually consists of testing specimen, loading control, as well as force and displacement measurement, etc. Analog to bulk mechanical testing, micro-scale mechanical testing also requires these essential components, which can be managed conveniently at micro- or even nano-scales thanks to the advancement of MEMS technologies. Some or all of the testing elements can be integrated with loading transducers and/or force/displacement sensors. We review 6 major micro-mechanical testing techniques, namely, nanoindentation, micro-tensile test, wafer curvature measurement, micro-compression test, bulge test, and micro-fatigue test.