Calphad 61 (2018) 219–226

Contents lists available at ScienceDirect

Calphad

journal homepage: www.elsevier.com/locate/calphad

Kinetic diffusion multiple: A high throughput approach to screening the composition-microstructure-micromechanical properties relationships

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ARTICLE INFO

Keywords:
Kinetic diffusion multiple
High-throughput methodology
Micromechanical testing technique
Electron back scattered diffraction (EBSD)
Composition-microstructure-micromechanical/properties relationships

ABSTRACT

We introduced a strategic "Kinetic Diffusion Multiple" (KDM) that undergoes interdiffusion annealing followed by realistic thermal treatment. The blended spectra of phases and microstructures subjected to treatment in deeply grooved composition gradients enables the microstructure and micromechanical properties of structural materials to be surveyed by high-spatially resolved micro-analysis along the composition arrays. The KDM was demonstrated as a robust high-throughput methodology that enables rapid screening of the composition-microstructure-micromechanical/properties relationships for different metallic materials. It has also proven great success at elucidating the lasting effects of alloying elements and diffusion flux on microstructure, micromechanical properties, phase transformation, and their interrelationships as a whole.

1. Introduction

The discovery, development, and optimization of today's new materials are facing three interrelated challenges [1], namely, advanced materials are often i) highly tailored, ii) formulated from multicomponents, and iii) exhibit intrinsic structure and behavior, thereby creating huge complex variable spaces to be explored. In years past, materials design significantly relied on a huge number of experiments for design, processing, and testing, largely by trial and error. Nowadays, the advances in computer science and modern characterization tools provoke knowledge-based materials design, which is placing unprecedented demands on the link between the associated variable space of target materials including composition, processing, microstructure and resultant mechanical properties.

In recent years, high throughput techniques have boosted acquiring the composition-microstructure link of materials for example by diffusion couple [2–6] as well as mapping the materials composition-properties link. Regarding the latter, a variety of experimental high throughput approaches have been developed that permit screening of the composition-property link in order to generate combinatorial libraries for a broad range of materials [7–9] such as thin film systems, functional materials such as shape memory alloys [10] and magnetic materials [11], and structural polymers [12]. As for bulk systems, the diffusion multiple approach, extended from the diffusion couple [13] and diffusion triple [14,15] techniques in metallurgy research, has been put forward to create the composition-property library (so far focused on thermo-physical properties including heat capacity, thermal conductivity and elastic constant, etc.), along with compositional phase diagram data, of ternary and multicomponent bulk metallic materials in a phase-based perspective [16,17].

Nevertheless, it is quite well known that in structural materials, not only by chemical composition, the mechanical behavior is to a large extent governed also by their microstructural attributes such as phase present and distribution and grain size at the scale of several nanometers and multiple micrometers, which define the microstructure-properties link. This microstructure-property link is essentially recognized as an indicator of heat/mechanical treatments, which consists of casting, heat/mechanical treatment and forming etc., conventionally applied by sequential fabrication/processing of homogeneous bulk specimens. This sequential complexity gives rise to difficulty in preparing high throughput bulk combinatorial samples of structural materials, thereby leaving conventional alloy discovery and development

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https://doi.org/10.1016/j.calphad.2018.04.001
Received 27 January 2018; Received in revised form 31 March 2018; Accepted 1 April 2018

largely one alloy at a time.

Nowadays, in view of the fact most of bulk (structural) materials are often both profoundly sensitive to chemistry and processing, there is a strong demand to delineate a full composition-microstructure-property link as both composition and microstructure are required to derive the resulting properties/performance. High throughput experimental technique is well suited to the task. This is particularly due to technical difficulties for any one-alloy-a-time approach (e.g. traditional equilibrated alloy method) in which requires obtaining realistic compositional homogeneity of target bulk materials and finding its genuine microstructure in response to the applied processing. This complicates determination of the outcomes from different experimental runs as functions of both compositions and microstructures, because associated effecting factors are difficult and/or costly to control. Innovative high throughput work to tackle these difficulties include: Gallant et al., via introducing thermo-mechanical gradients and surveying the processing parameters that influence bulk energy materials [18], in situ screening that acquired the evolution of phases associated with gas-solid reactions in combinatorial catalyst libraries [19], and the process-property linkage of structural Al-6061 alloy [20].

Meanwhile, promising high throughput diffusion studies have focused on materials subjected to either diffusion annealing or processing by a single stage that essentially generates a single array, either continuous or discrete, of compositions or processing of materials without further treatments, thus rendering it a one-array-at-a-time approach. New advance in bulk materials were suggested to envisage freezing the spinodal decomposition of a hypothetical binary system [21], to scan the mechanical properties versus nine ageing treatments over 5 semi-continuously processed triplex steels with varied compositions [22], and to investigate phase diagrams and massive phase precipitation in the Fe-Cr-Ni system by a dual-anneal diffusion multiple approach [23]. Nevertheless, there is not yet a method to map the microstructural and mechanical properties for solid-state bulk materials resulting from actual treatment routes or kinetic process.

To face these challenges, we proposed a strategic high throughput methodology, called as kinetic diffusion multiple (KDM), that undergoes interdiffusion annealing to generate continuous composition gradients (particularly suitable at a single-phase region/temperature in order to avoid any extra influence and complexity of possible phase transformation), followed by subsequent realistic thermal treatments like solutioning and/or ageing to trigger favorable phase transformation(s). The resultant blended spectrum of phases and microstructures in the established composition gradients in the KDM will be locally characterized and surveyed by high-spatially resolved electron probe microanalysis (EPMA) and electron back-scattered diffraction (EBSD) while the micromechanical properties can be screened over the established (i.e. indented) composition arrays by advanced micro-nano mechanical testing techniques. Our approach is schematically shown in Fig. 1 together with the conventional diffusion multiple [16].

2. Material and experimental methods

2.1. Preparation and treatments of KDM

2.1.1. KDM of quinary Ti alloys

The KDM of quinary titanium alloys, consisting of pure-Ti and the Ti-7.73Al-8.44 V, Ti-7.72Al-8.01Cr and Ti-13.4Al-12.4Mo (wt%) alloy blocks, was designed with its assembled configuration depicted in Fig. 1b. Each composition in the multiple and its position in the assembly were determined to allow a specific diffusion flux between different alloying elements. All four component blocks were machined via electrical discharge machining (EDM) from ingots prepared by arc melting using 99.9% sponge Ti, 99.99% Al, 99.99% Cr, 99.99% V and 99.9% Mo according to alloy composition, in an argon atmosphere. To achieve a homogeneous composition, arc melting was repeated ten times for individual ingot samples, and then all ingots were homogenized at 1473 K for 12 h. The contact surfaces were grinded and polished in a conventional metallographic way. The assembled blocks were then bonded in a vacuum diffusion bonding machine at 1273 K for 2 h with a load of 15 MPa to fabricate the diffusion multiples. The diffusion multiples were then annealed at 1473 K for 12 h in vacuum followed by furnace cooling to produce wide diffusion zones. To develop the phase and microstructure across the composition gradients, a processing route of aging in the α + β region of the phase diagram at 1273 K and subsequent cooling was performed.

2.1.2. KDM of Ti-Al-Mo ternary

Two Ti-Al-Mo ternary KDMs, Ti/Ti-7.58Al-4.97Mo (at%) and Ti-1.52Mo/ Ti-5.04Al (at%), were prepared. The fabrication of the KDM was the same as above, except for a back-sealing into quartz capsules before the BSA treatment to induce the β to α phase transformation process. Considering the α/β phase transformation temperatures of individual end-member alloys, the solid-solution treatment of the Ti/Ti-7.58Al-4.97Mo and Ti-1.52Mo/Ti-5.04Al KDMs were performed at 1193 K and 1253 K for 15 min respectively.

2.1.3. KDM of Mg-Zn binary

The Mg-2.3 at%Zn alloys were prepared by melting commercially pure Mg and Zn in a steel crucible coated with boron nitride, enclosed in a vacuum induction melting and casting system (VSG 002 DS, PVA TePla) under a protective atmosphere of Ar. The pure Mg and Mg-Zn alloys were then subjected to homogenization at 673K for 360 h. The rods were then sectioned into discs of 8 mm in thickness and 12 mm in diameter. The assembled bulks were then diffusion bonded by a Physical Simulator (GLEEBLE 3800, DSI) at 400 ℃ 673 K for 1 h with a load of 17.6 MPa to fabricate the KDM. The bonded diffusion couples were then annealed at 723 K for 184 h after encapsulation in pyrex tubes under an argon atmosphere with the aim of preventing oxidation.

2.1.4. KDM of Co-Al-V ternary

The Co-Al-V alloy ingots were prepared from 99.95% Co , 99.99% Al and 99.9% V(mass%) by arc melting in an argon atmosphere. The arc melting was repeated at least ten times to guarantee compositional homogeneity. All the ingots were annealed at 1523 K for 12 h under vacuum, resulting in coarse grain size (typically larger than 1 mm). Cylinders of φ12 × 5 mm were cut from these alloy ingots and pure Co bulks. The well-contacted cylinders were assembled and bonded at 1223 K for 90 min with a load of 10 MPa. Then the bonded diffusion couples were sealed into evacuated and argon-back-flushed quartz capsules, followed by diffusion annealing at 1373 and 1473 K for 24 h and 12 h, respectively, with quenching into ice water.

2.2. Microanalytic characterization of KDM

2.2.1. Compositional mapping

Following conventional metallographic preparation, an electron probe micro-analyzer (EPMA, JEOL JXA 8900) was employed to measure and map the local composition of diffusion zones with particular emphasis in the center and four interfacial areas in the multiple. Only the EPMA data with a total weighigh throughput percentage between 99% and 101% were selected for this work.

2.2.2. Microstructural examination

After thorough polishing, Ti alloy multiples were etched in Kroll's reagent (2 vol% HF + 6 vol% HNO_3 in water) for 15 s. The optical microscope (OM) and scanning electron microscope (SEM, EVO MA15 Zeiss) were employed to characterize the variations of microstructures with various compositions in multiple.

2.2.3. Crystallographic analysis

KDMs were prepared for EBSD analysis by manually mirror polishing with a series of diamond polishing pastes of progressively