Powder Diffusion Multiple Approach for High Throughput Exploration of High Entropy Alloys for High Temperature and Nuclear Applications

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1. Introduction

The innovative materials are required to serve in the severe service conditions of forthcoming nuclear power plants having uprated power and prolonged service life. The new materials with enhanced properties can also aid in life extension of existing nuclear power plants by ensuring materials integrity. The conventional alloy design strategy i.e. one-alloy-at-a-time requires greater number of samples and long time. The extensive efforts are also required to minimize contamination during various stages of experiments [1]. The high melting point of refractory metals make the conventional approach more tedious due to the difficulty in melting and achieving homogeneity [2].

In contrast to the conventional alloy development methods, the high throughput alloy exploration, in which a library of various alloy compositions is developed in just one or few sample, is gaining more and more research interest [3]. The high throughput alloy exploration are being reported via diffusion multiples developed by assembling different metal blocks [3]. The intimately connected metal blocks are heated to form the solid solutions via interdiffusion [4]. Fig. 1 illustrates a typical diffusion multiple comprising of blocks of metals A, B, C, D and E.

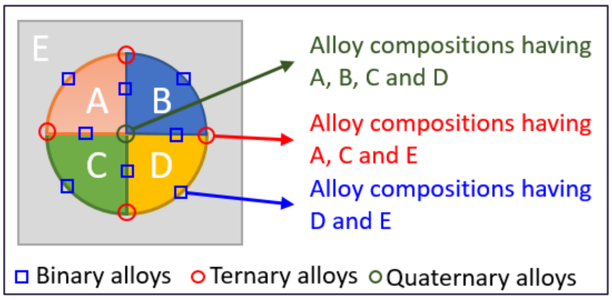

Fig. 1. Diffusion multiple made up to metal blocks.

The interdiffusion among metallic blocks produces several phases having various compositions, which are characterized by using microscale examinations [3]. However, the monoblocks-based diffusion multiple shows limited number of junctions as shown in Fig. 1 and hence a little number of alloy compositions are possible.

In order to increase the possible number of alloy compositions in a single diffusion multiple, we are presenting novel powder diffusion multiple approach. After characterizing the alloy compositions (solid solution phases) developed in powder diffusion multiple, the solid solution with homogeneous structure and promising properties can be selected. Large amount of the selected solid solutions can be produced by using conventional metal/alloy making technologies to fulfill the industrial needs.

2. Methods and Results

2.1 Experimental

In order to develop powder diffusion multiples, granules of a pure metal (any one of Ti, Cr, Mo, Nb, Al and Zr) were packed in the manually blended mixture of metallic powders (all other than granules). The configuration of powder diffusion multiple is shown in Fig. 2.

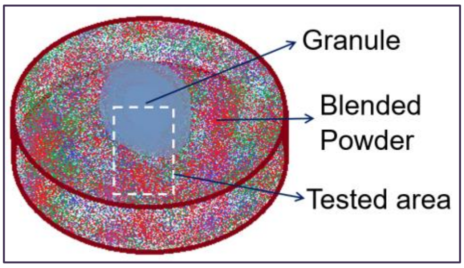

Fig. 2. Schematic of powder diffusion multiple having granules (e.g. Ti) surrounded by powder mixture (Cr, Mo, Nb, Al and Zr).

After spark plasma sintering of diffusion multiple at 1500 ºC for 5 minutes, the microstructural characterization was carried out by scanning electron microscope (SEM) and Energy Dispersive Spectroscopy (EDS). In order to analyze the variation in mechanical behavior with varying composition, micro-Vickers hardness tests with 500g force and a 10-second dwell time were carried out over ~3x3mm^2 of test area, keeping the distance between consecutive test points more than 5 times of the indent's diagonal length.

2.2 Results

The diffusion multiple exhibited compositional gradients and multiple phases enrich different constituents. The representative microstructure of a diffusion multiple i.e. AlZrNbMoCr (powder)-Ti (granules) is shown in Fig. 3.

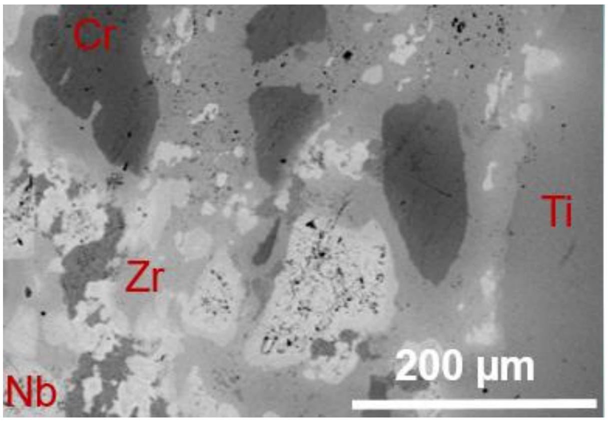

Fig. 3. SEM microstructure of AlZrNbMoCr (powder)-Ti (granules) showing Cr-, Zr-, Nb- and Ti-rich phases.

In order to check the variation in hardness with varying composition, the polished surface of the diffusion multiple was examined by Vickers hardness test, and hardness contour were developed, as represented in Fig. 4.

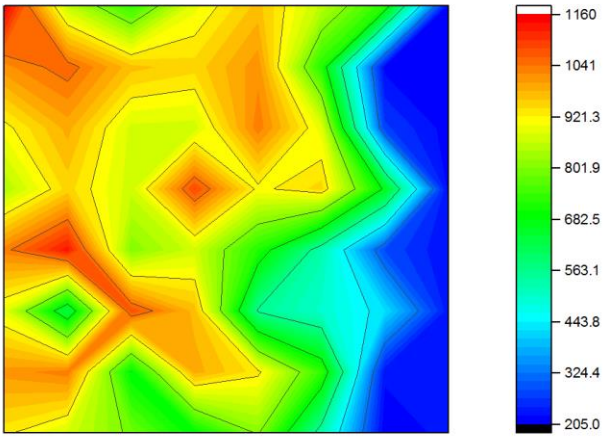

Fig. 4. The hardness contours of of AlZrNbMoCr (powder)-Ti (granules) samples.

The diffusion multiple shows varying hardness due to multiple phases with varying compositions. The minimum and maximum hardness in AlZrNbMoCr (powder)-Ti (granules) sample are 205 HV and 1160 HV, respectively. The hardness values were related to the composition of the alloys through point EDS analysis on the indents. Depending upon the higher hardness, following six HEA-like compositions have been shortlisted, as shown in Table I.

Table I: HEA-like compositions explored via powder diffusion multiple

Composition (at.%)	Hardness (HV)
$Al_{22.9}Zr_{34.1}Nb_{4.3}Mo_{11.7}Ti_{17.3}Cr_{9.8}$	~1480
$Al_{50.6}Zr_{20.7}Nb_{8.0}Mo_{0.3}Ti_{5.8}Cr_{14.6}$	~1340
$Al_{40.7}Zr_{34.7}Nb_{6.5}Mo_{5.8}Ti_{4.1}Cr_{8.2}$	~1240
$Al_{35.6}Zr_{11.2}Nb_{12.8}Mo_{37.1}Ti_{1.2}Cr_{2.1}$	~1190
$Al_{49.0}Zr_{10.0}Nb_{19.4}Mo_{0.3}Ti_{8.4}Cr_{20.9}$	~1190

Al is known to have low inherent density and BCC stabilizing characteristics [5]. Therefore, the selected HEA-like compositions which have at least ~30at.% Al (Table I), show high strength to weight ratio, as shown Fig. 5.

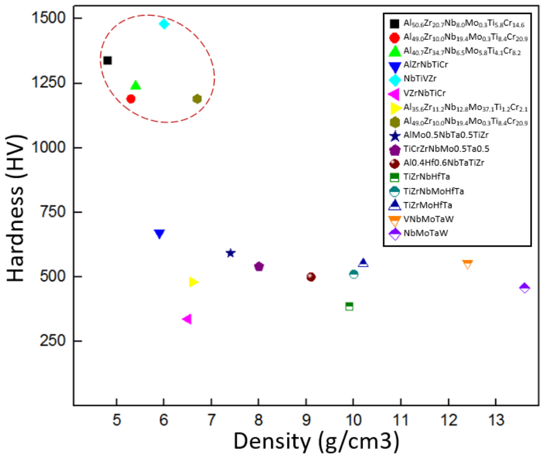

Fig. 5. The comparison of hardness and theoretical density (strength to weigh ratio) of selected HEA-like compositions explored through powder diffusion multiple with several HEAs.

Although the high strength to weight ratios of selected-HEA like compositions shown their usefulness for nuclear applications, but it is necessary to verify these results by preparing bulk samples having this compositions. Moreover, the high temperature mechanical properties is also worth to analyze in order to ensure high temperature and nuclear applications.

3. Conclusions

The development of powder diffusion multiple by spark plasma sintering produced multiple phases. The compositional analysis and micro hardness tests on the same points revealed several HEA-like compositions with high strength to weigh ratio, which are to be analyzed by producing bulk samples.

Acknowledgment

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