



# Effect of alloying order on non-isothermal sintering kinetics of mechanically alloyed high entropy alloy powders

Rahul B. Mane, Bharat B. Panigrahi\*

Department of Materials Science and Metallurgical Engineering, Indian Institute of Technology Hyderabad, Kandi, Sangareddy 502285, Telangana, India



## ARTICLE INFO

### Article history:

Received 11 December 2017  
Received in revised form 12 January 2018  
Accepted 15 January 2018

### Keywords:

High entropy alloy  
Mechanical alloying  
Sintering  
Diffusion

## ABSTRACT

This work correlates the densification behaviors of high entropy alloy powders with the alloying order. Mechanically alloyed powders (CoFeNi, CoFeNiCr, CoFeNiCrMn and AlCoFeNiCrMnAl alloys) were non-isothermally sintered using dilatometer. It was demonstrated that when the alloying order or the number of alloying elements were increased from 3 elements to 6 elements, sintering kinetics were changed. Materials exhibit sluggish grain growth, reducing densification rates and increasing activation energies of sintering, with increasing configurational entropy. Increasing trend of micro-hardness also indicates the better mechanical properties of such alloys.

© 2018 Elsevier B.V. All rights reserved.

## 1. Introduction

High entropy alloys (HEA) are among the most studied topics, since last decade [1,2]. It was observed that with increasing number of elements, several properties of the alloy were also changed [1]. For example, hardness was reported to be increased from 1.47 GPa to 2.45 GPa and 10.14 GPa with increasing elements from CoFeNi alloy [3] to CoCrFeNi [4] and CoCrFeNiAl [5] alloys respectively. Young's modulus was improved from 162 GPa to 233 GPa when the composition of the alloy was changed from CoFeNi to CoCrFeNi [5]. Tracer diffusion method and diffusion couples method in multi-component alloys were reported, where diffusion characteristics were found to be mostly slower in nature [2,5–8].

Powder sintering is one of the major fabrication routes [2,4,9], however, the understanding of sintering mechanism has been rarely addressed. Since diffusivity is affected by alloying in HEA, and sintering may also be affected. The present study aims to understand the effect of the alloying order, i.e., the sequential increment in the composition from 3 elements to 6 elements, such as CoFeNi, CoFeNiCr, CoFeNiCrMn and AlCoFeNiCrMn alloys; on the densification kinetics of the powders, during non-isothermal heating.

## 2. Materials and methods

Elemental powders (-325 mesh) of about 99.5% purity were mixed in an equimolar ratio for respective CoFeNi (3E), CoFeNiCr (4E), CoFeNiCrMn (5E) and AlCoFeNiCrMn (6E) alloys. Powder mixtures were milled on a planetary ball mill (Fritsch P-5) using WC balls and vials with toluene media, for about 15 h at a speed of about 300 rpm; where ball to powder weight ratio was about 15:1. Small cylindrical pellets (green density of about 60%) were prepared at a uniaxial pressure of about 70 MPa. Green compacts were heated non-isothermally (at a rate of 10 °C/min up to about 1150 °C under purified Ar), on a vertical dilatometer system (Theta Inc. USA). The densities were measured using Archimedes principle. Hardness was measured using Vickers hardness tester (Dura Scan, EMCO TEST). Samples were characterized using X-ray diffraction (Cu  $\alpha$  radiation), scanning electron microscope (FESEM, ZEISS) and transmission electron microscope (JEOL-2100 TEM).

## 3. Results and discussion

### 3.1. Synthesis

XRD patterns of as-milled powders and sintered samples (3E, 4E and 5E) show (Fig. 1) mostly FCC phase, whereas 6E powder consists of both FCC and BCC phases. Very small peaks of sigma phase were also observed in all the samples. After sintering, 6E alloy was

\* Corresponding author.

E-mail address: [bharat@iith.ac.in](mailto:bharat@iith.ac.in) (B.B. Panigrahi).

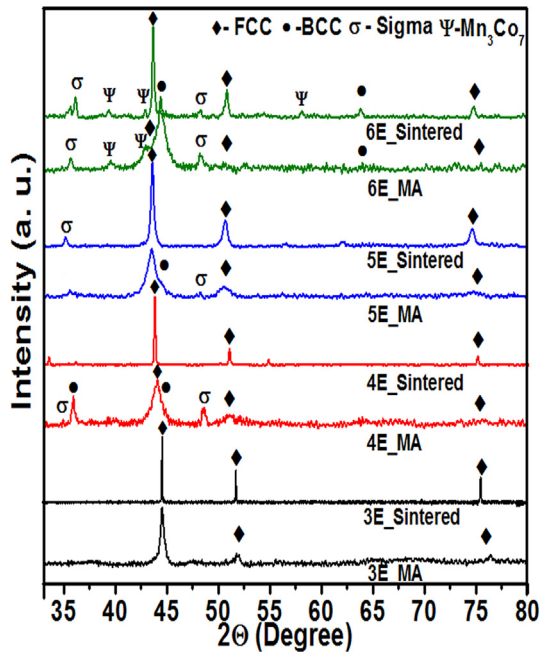


Fig. 1. XRD patterns of as-milled powders & sintered samples of 3E, 4E, 5E and 6E alloys.

also found to exhibit FCC as a major phase and BCC as a minor phase. SEM micrographs of as-milled powders (Fig. 2a–d) show the large agglomerates, spread over about 10 to 20  $\mu\text{m}$ , but particles seem to be highly irregular in shape. TEM images with diffraction patterns (mostly FCC type in 3E, 4E and 5E and BCC type in 6E) were also shown (Fig. 2); where grains were typically smaller than 200 nm.

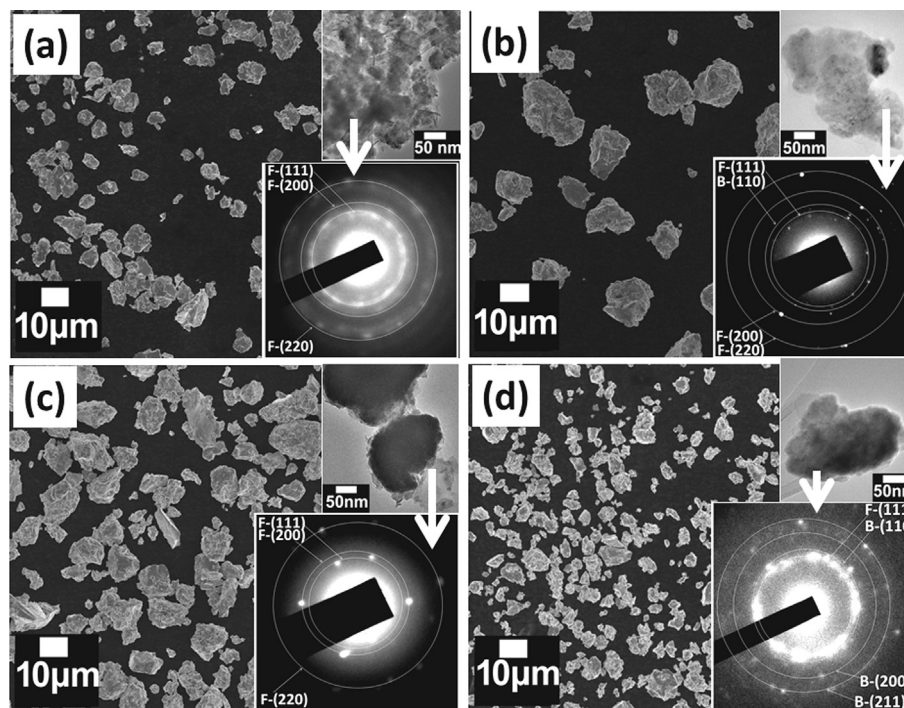


Fig. 2. SEM and TEM micrographs (with respective diffraction patterns) of as-milled powders for: (a) CoFeNi, (b) CoCrFeNi, (c) CoCrFeNiMn, and (d) AlCoCrFeNiMn alloys.

### 3.2. Sintering

Shrinkage recorded as a function of homologous temperature ( $T/T_m$ ; where  $T$  is sintering temperature) have been shown in Fig. 3a. Melting points ( $T_m$ ) of the alloys were determined separately through differential scanning calorimetry; and were found to be 1310  $^{\circ}\text{C}$ , 1290  $^{\circ}\text{C}$ , 1245  $^{\circ}\text{C}$  and 1240  $^{\circ}\text{C}$  for 3E, 4E, 5E and 6E alloys respectively. Little enlarged view of the dilatometer curves of 4E, 5E, and 6E samples have been shown in the inset of Fig. 3a; which show the decreasing trend of shrinkage with increasing elements. The axial shrinkages derived from the dilatometer plots by usual method [10] have been shown in Fig. 3b. It was found that 3E sample showed higher shrinkage (Fig. 3b); however, the shrinkage was found to be drastically decreased when alloying numbers were increased to 4 or higher. 3E sample exhibited higher relative density ( $\sim 85\%$ ) compared to that of 6E sample ( $\sim 72\%$ ) after sintering (Table 1).

Sintering is a complex process where several mechanisms may contribute simultaneously. Mechanically alloyed powders often contain, meta-stable phases and sigma phases etc. along with major phase, which may also provide additional driving forces during sintering. However, their contribution in densification is often limited and often some diffusional mechanism, i.e., grain boundary diffusion (GBD) or volume diffusion (VD), dominates the process over others. Axial shrinkage data were analyzed through the widely used non-isothermal sintering model [11]:

$$\ln[T^p(dY/dT)] = \ln C - Q/[(n+1)RT] \quad (1)$$

where,  $dY$  is  $\Delta L/L_0$ ,  $L_0$  is the initial length,  $\Delta L$  is the change in length,  $T$  is the temperature,  $R$  is the universal gas constant,  $C$  is a constant.  $P$  has the value of  $3/2$  for VD and  $5/3$  for GBD, and the values of  $n$  are 1 and 2 for VD and GBD respectively [11]. Activation energies ( $Q$ ) of sintering were estimated through Arrhenius plots as shown in Fig. 3c & d; and also shown in Table 1.  $Q$  was found to increase with increasing alloying order.

Download English Version:

<https://daneshyari.com/en/article/8014281>

Download Persian Version:

<https://daneshyari.com/article/8014281>

[Daneshyari.com](https://daneshyari.com)