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XRD analysis of High Entropy Alloys $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ and $Si_{0.1}(CoCrCuFeNi)_{0.9}$

Abhishek Saurav[1], G Kushal Sai[1], J Lakshmi Narasimha[1], Vamsi Krishna K[2], M Satya Prasad[1] and Balaji Rao Ravuri[2,*]

[1] Department of Aerospace Engineering, GITAM Deemed to be University, Hyderabad, Telangana, 502329, India

[2] Department of Physics, GITAM Deemed to be University, Hyderabad, Telangana, 502329, India

Abstract

High Entropy Alloys $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ and $Si_{0.1}(CoCrCuFeNi)_{0.9}$ were synthesized using the popularly identified mechanical alloying method - The Ball Milling method which happens to be a easily accessible method to convert bulk powder to Nano powder. Both the compounds were calculatively weighed-in; the vacuum chamber which had Argon atmosphere inside it. The compounds were ball milled for 15 hours and 30 hours. XRD tests were performed through which the particles lattice strain and particle size were obtained proving that the end product obtained is indeed a nano powder.

Keywords: Mechanical alloying, XRD, Lattice strain

1. Introduction

In High entropy alloys (HEAs) are deemed to be the very recent classification of materials showing excellent mechanical properties for their applications in the field of compound structures and transportation [4]. They usually are defined as alloys that are formed by mixing equal or relatively large proportions of (usually) five or more elements [2]. Equiatomic substitution plays the primary role in the formation of HEA's in which the replacement of individual components with multi-component equiatomic or near-equiatomic mixtures of chemically similar species takes place [7]. It is found that some HEAs have considerably better strength-to-weight ratios, with a higher degree of fracture resistance, tensile strength, as well as corrosion and oxidation resistance than conventional alloys [9]. They are known to increase the energy efficiency in various applications especially in the field of aerospace, materials used as modern engine components must be able to withstand extreme operating temperatures, creep, fatigue crack growth and translational movements of parts at high speed. Therefore, the parts produced must be lightweight and have good elevated temperature strength, fatigue, resistant to chemical degradation, wear and oxidation resistance [8].

These materials were designated as "High-entropy alloys" to signify their entropy increase of mixing which is substantially higher when there is a larger number of elements involved in the mix, and their proportions are more nearly equal [3]. These alloys are currently in the limelight in the field of materials science and engineering because they have exclusive potentially desirable properties [1]. Furthermore, research indicates that some HEAs have considerably better strength-to-weight ratios, with a higher degree of fracture resistance, tensile strength, as well as corrosion and oxidation resistance than conventional alloys [1,2,6,7]. The key factor in HEA's is that each principal element should have a concentration between 5% and 35% [6].

Corresponding Author E-mail: ravuri309185@gmail.com

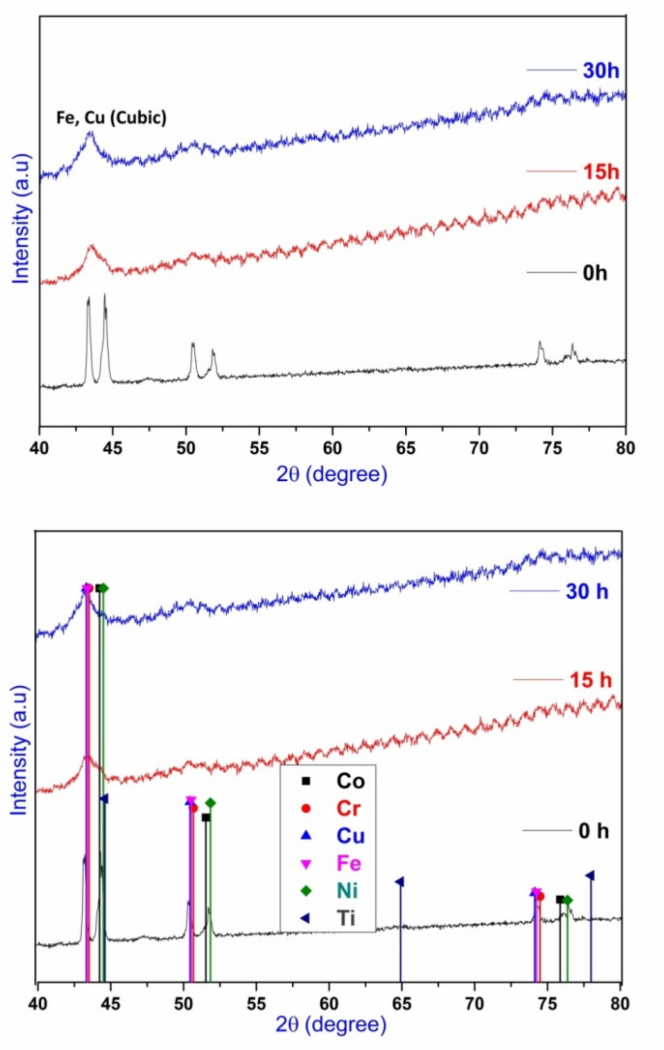

Fig. 1. The X-ray diffraction patterns of Ti$_{0.1}$(CoCrCuFeNi)$_{0.9}$ alloys for 0, 15 and 30 h Ball Milling.

2. Experimental details

The initial preparation of the samples the weighing in procedure and calculations were given utmost priority and all the raw materials chosen was of AR grade which had the maximum purity of 99.9%. All the samples were taken in individually in the vacuum chamber which had inert gas atmosphere inside it. All the samples were weighed in calculatively considering the concept of atomic weight ratio using cross multiplication method. The samples were further mixed initially using hand grinding method using pestle and mantle for 30 minutes.

The stainless steel vial of the ball milling machine were initially cleaned with sand paper and then with acetone to assure the absence of impurities in the sample vials. The compounds

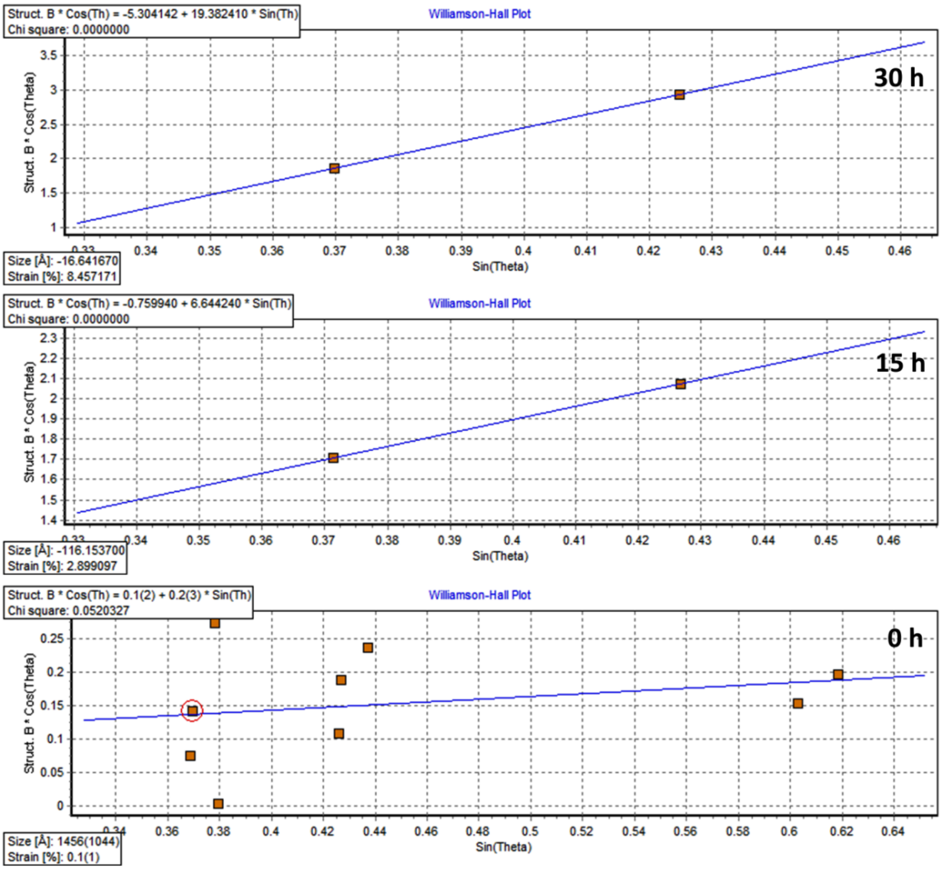

Fig. 2. Williamson–Hall plots of $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ alloys ball milled for 0,15 and 30 h

Table 1. The crystallite size and lattice strain of $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ alloys ball milled for 0h, 15h and 30 h respectively.

Milling Time	Crystallite size (nm)	Lattice strain (%)
0 hours	145.6	0.1
15 hours	11.61	2.899
30 hours	1.6	8.457

were further subjected to ball milling for 15 hours and 30 hours respectively. The weight of the balls was carefully taken in to maintain the ionization ratio 1:10. Special care was taken while programming the ball milling machine.

3. Results and discussion

XRD technique is the most widely employed technique to trace the nature of the bulk material such as amorphous, polycrystalline and crystalline nature. It enables us to identify the material, determine its crystal structure and degree of crystallinity, obtain the size of crystallites, the unit cell size, etc. All glass and glass-ceramic samples are crushed into fine powders and is further used to determine the XRD patterns. The amorphous and crystallization phase details are studied by using PAN analytical Diffractometer B.V fitted with Cu target (Kα wavelength of 0.154 nm) and Ni filter at 40 kV and 30 mA (range:40-80° with a step size of 0.05°) at room temperature.

The crystallite size and lattice strain of the alloys can be calculated from peak broadening of XRD using Scherer formula and Williamson–Hall analysis, given below in Equations (1) and (2).

$$D = \frac{0.94\lambda}{\beta cos\theta} \qquad (1)$$

$$\beta cos\theta = \frac{c\lambda}{D} + 4\varepsilon Sin\theta \qquad (2)$$

Where, β, θ, C, D, ε and λ are the FWHM, Bragg angle, correction factor (C ≈ 0.94), size of crystallites (nm), lattice strain, and the wavelength (0.154 nm) of X-ray, respectively. Below are the graphical results of the samples $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ and $Si_{0.1}(CoCrCuFeNi)_{0.9}$ respectively.

Figure 1. shows the powder XRD profile of $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ alloys for the ball milling time of 0h, 15h and 30h. The detailed phases, crystallite size and lattice strain are identified by using X'Pert High Score plus software (PANAnalytical). All peaks in XRD are well indexed with the given parametric ranges provided by the software library and no further impurity phase was identified. All major crystalline peaks were formed between the angle 40-80° range. At 0h ball milling all crystalline peaks are well indexed with Co, Cr, Fe, Ni, Cu and Ti phases. Further, as the milling time increased from 0h to 15h - 30h, all crystalline peaks are minimized and confirmed only Cu and Fe crystalline phases. However these Fe and Cu phases have cubic structure which is most stable and favourable mechanical and magnetic properties to the HEAs (Fig.1.). From Fig.2. it is observed that lowest crystalline size ~1.6 nm and highest lattice strain 8.457% for $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ HEAs at 30 h ball milling.

Table 1. shows the crystallite size and lattice strain of $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ alloys ball milled for 0h, 15h and 30 h respectively. Williamson-Hall (W-H) analysis is a simplified integral breadth method where both size-induced and strain-induced broadening, de-convoluted by considering the peak width as a function. It is usually employed for estimating crystallite size and lattice strain, although the XRD analysis is an average method, they still hold utmost priority for grain size determination, apart from TEM micrographs

Figure 3. shows the powder XRD profile of $Si_{0.1}(CoCrCuFeNi)_{0.9}$ alloys for the ball milling time of 0h, 15h and 30h. The FWHM, Bragg angle, correction factor (C ≈ 0.94), size of crystallites (nm) and the lattice strain are done using X'Pert High Score plus software (PANAnalytical). All peaks in XRD are well indexed with the given parametric ranges provided by the software library and no further impurity phase was identified. All major crystalline peaks were formed between the angle 30-80° range. We can further observe the least crystallite size to be 0.61nm and the lattice strain to be 14.6% shown in table 2., which depicts the crystal imperfections as a sum result of the distribution of the lattices obtained.

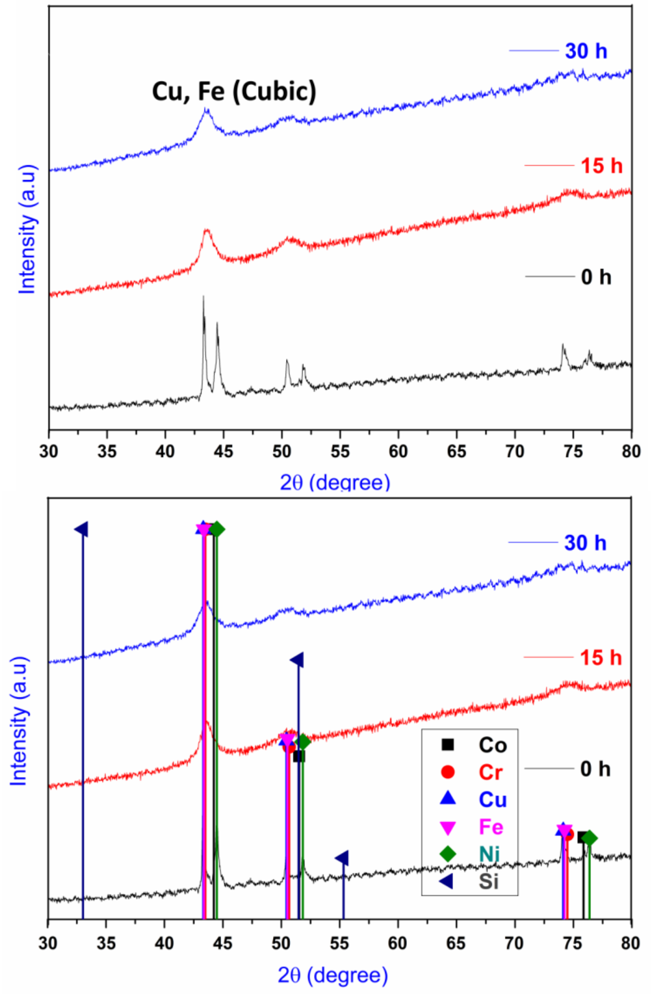

Fig.3. The X-ray diffraction patterns of $Si_{0.1}(CoCrCuFeNi)_{0.9}$ alloys for 0h, 15h and 30h Ball Milling

4. Conclusion

From the obtained results it can been concluded that, all major crystalline peaks were formed between the angle 40-80° range and 30-80° range for $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ and $Si_{0.1}(CoCrCuFeNi)_{0.9}$ compounds respectively. For the compound $Ti_{0.1}(CoCrCuFeNi)_{0.9}$ it is observed that lowest crystalline size ~1.6 nm and highest lattice strain 8.457% for 30h ball milling and for the compound $Si_{0.1}(CoCrCuFeNi)_{0.9}$ it can be observed that lowest crystalline size ~0.61 nm and highest lattice strain 14.6% for 30 h ball milling, by which it can be concluded that the bulk powder has successfully been converted to the lest forms of its nano-powder form.

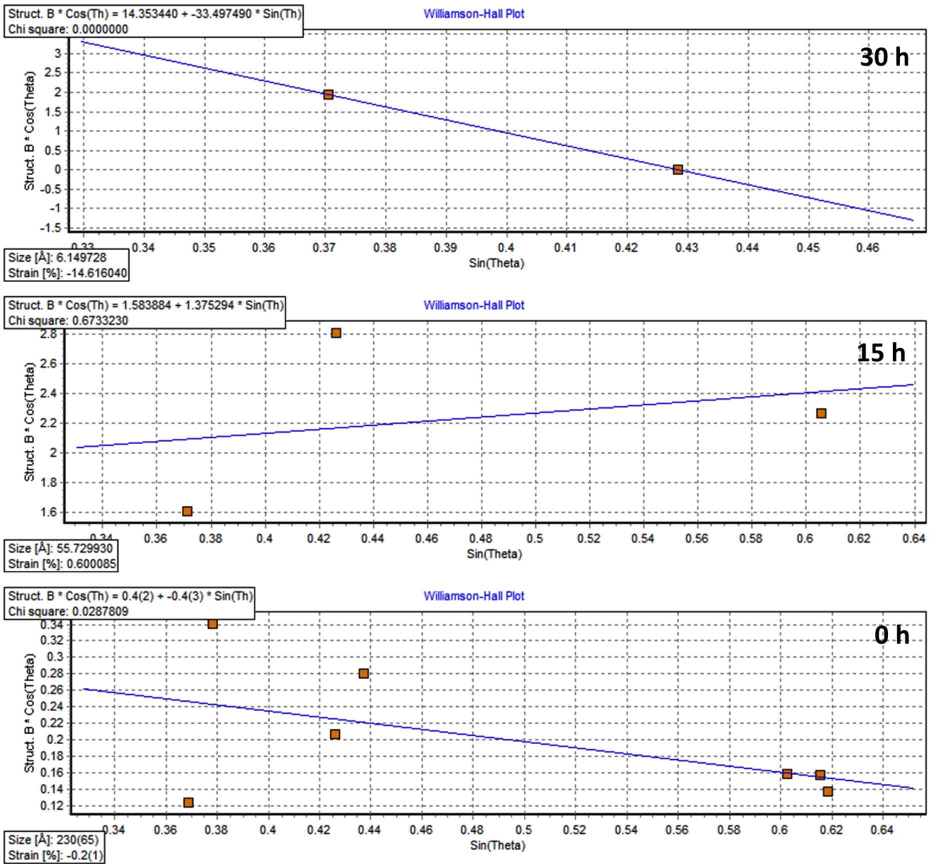

Fig.4. Williamson–Hall plots of $Si_{0.1}(CoCrCuFeNi)_{0.9}$ alloys ball milled for 0h,15h and 30h

Table 2. The crystallite size and lattice strain of $Si_{0.1}(CoCrCuFeNi)_{0.9}$ alloys ball milled for 0h, 15h and 30 h

Milling Time	Crystallite size (nm)	Lattice strain (%)
0 hours	23	0.2
15 hours	5.5	0.6
30 hours	0.61	14.6

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