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Synthesis And Characterization Study Of Alcrnifezn High Entropy Alloy Powders Through Mechanical Alloying

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High-entropy alloy powders with a nominal composition of AlCrNiFeZn were synthesized through 30 hours of mechanical alloying (MA). High-energy ball milling was performed using tungsten carbide vials and balls. The morphological evolution of the initial elemental powders and the high entropy alloy powders at various milling durations (0, 5, 10, 20, and 30 hours) was analyzed using Scanning Electron Microscopy (SEM). As the milling time increased, the powder particles underwent repeated plastic deformation, fracturing, and cold welding, leading to significant morphological changes. After 30 hours of milling, the powders exhibited nearly equiaxed and spherical particles. The elemental composition of the powders was confirmed using Energy Dispersive Spectrum (EDS) analysis. The crystallite size and lattice strain of the synthesized HEA were examined through X-ray Diffraction (XRD). After 30 hours of mechanical alloying, the crystallite size was found to be 45 nm, and the lattice strain was 0.65%. The lattice parameter was determined to be 3.0435 Å. The microstructure of the HEA revealed a dual-phase structure, consisting of an ordered body-centered cubic (BCC) phase and a soft face-centered cubic (FCC) phase.

Keywords: High Entropy Alloy: Mechanical Alloying: X-Ray Diffraction; Scanning Electron Microscope.

INTRODUCTION

High-entropy alloys (HEAs) have garnered significant attention over the past two decades due to their exceptional mechanical properties and adaptability to various service environments. Since the concept of high entropy alloy was first introduced in 2004 [1], extensive research has explored numerous elemental combinations, as these alloys demonstrate excellent mechanical strength, high-temperature stability, wear resistance, and corrosion resistance.

High entropy alloys have been developed using various synthesis methods, including stir casting [2], laser cladding [3], vacuum arc melting [4], gas atomization [5], and mechanical alloying (MA) [6]. Among these, mechanical alloying offers distinct advantages, such as uniform dispersion of components and superior chemical homogeneity [7–11]. In a previous

study, $Al_{10}Cr_{25}Co_{20}Ni_{25}Fe_{20}$ High entropy alloy were synthesized through mechanical alloying, followed by conventional and spark plasma sintering [6]. There is limited work available of equimolar high entropy alloy fabricated through mechanical alloying and its characterization study. There is always need of new composition of HEAs with superior mechanical properties and its characterization analysis in aerospace and automotive industries for high temperature applications.

In the present research, AlCrNiFeZn equimolar HEA powders were synthesized using 30 hours of mechanical alloying. The morphological evolution and characteristics of the synthesized powders were analyzed using various advanced characterization techniques.

Materials and Methods

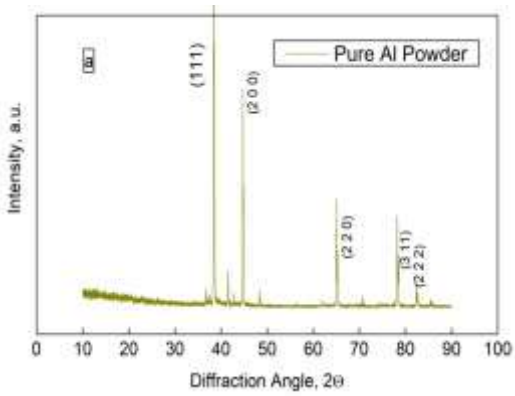
High-purity elemental powders (>99%) of aluminum (Al), chromium (Cr), nickel (Ni), iron (Fe), and zinc (Zn), with particle sizes of 40 microns (-325 mesh size), were used as raw materials. Toluene was employed as a process control agent during the milling process. The powders were mixed in tungsten carbide vials with tungsten carbide balls. The ball milling was conducted at a speed of 300 rpm, with a ball-to-powder ratio (BPR) of 10:1. To prevent overheating of the vials, the milling process was interrupted every 20 minutes, with a 10-minute cooling period.

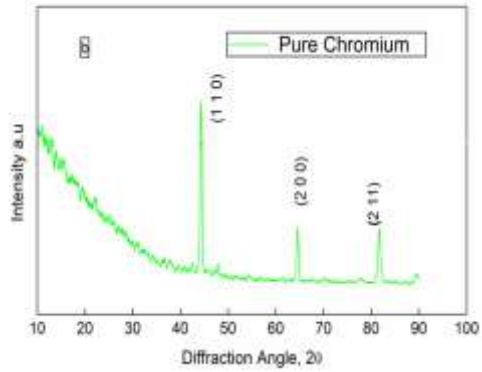
The milled powders were collected at intervals of 0, 5, 10, 20, and 30 hours for characterization. The morphological and chemical composition analysis of both the initial pure powders and the mechanically alloyed high entropy alloy powders at different milling times (0, 5, 10, 20, and 30 hours) was performed using a Scanning Electron Microscope (SEM, JEOL JSM).

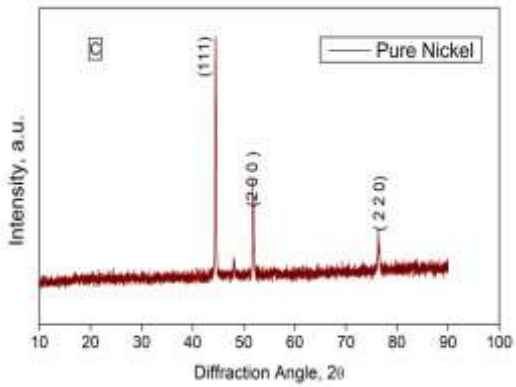
The phase constitution of the prepared High entropy alloy was determined using X-ray Diffraction (XRD) analysis (Rigaku Ultima III), which also provided insights into the crystallite size, lattice strain, and lattice parameter of the alloy. X-ray diffraction measurements were carried out using Cu-Kα radiation, with a scanning speed of 3°/min over a 2θ range of 10°–80°.

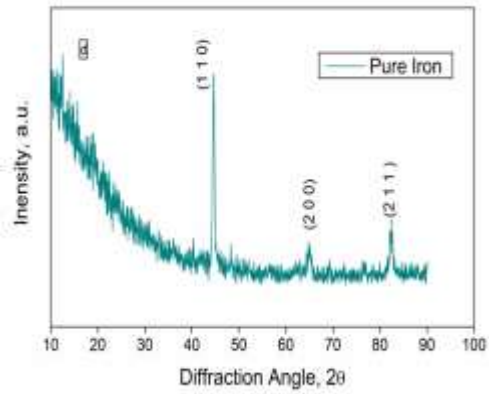
Result and Discussion

Fig. 1(a)-(e) shows the X-ray diffraction patterns of the pure Al, Cr, Ni, Fe, and Zn powders. For aluminum, five major peaks were observed corresponding to the (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) planes.









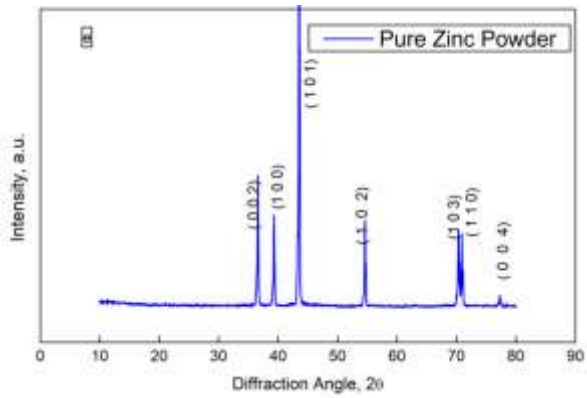


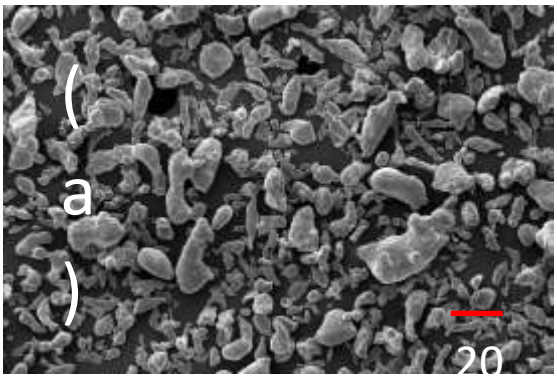
Fig.1 shows the X-ray diffraction images of as received pure (a) Al, (b) Cr, (c) Ni, (d) Fe and (e) Zn powders.

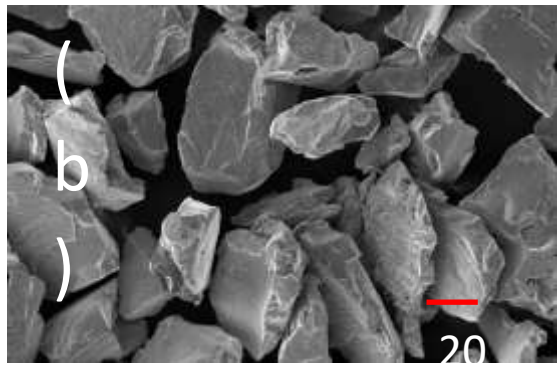
Chromium exhibited peaks corresponding to the (4 0 0), (0 0 4), (0 0 2), and (2 1 1) planes. For nickel, the peaks observed were (1 1 1), (2 0 0), and (2 2 0), while iron displayed peaks for the (1 1 0), (2 0 0), and (2 1 1) planes. Zinc showed peaks corresponding to the (1 0 2), (1 0 0), (1 0 1), (1 0 2), (1 0 3), (1 1 0) and (0 0 4) planes. These peaks confirmed the purity and crystallographic nature of the raw powders.

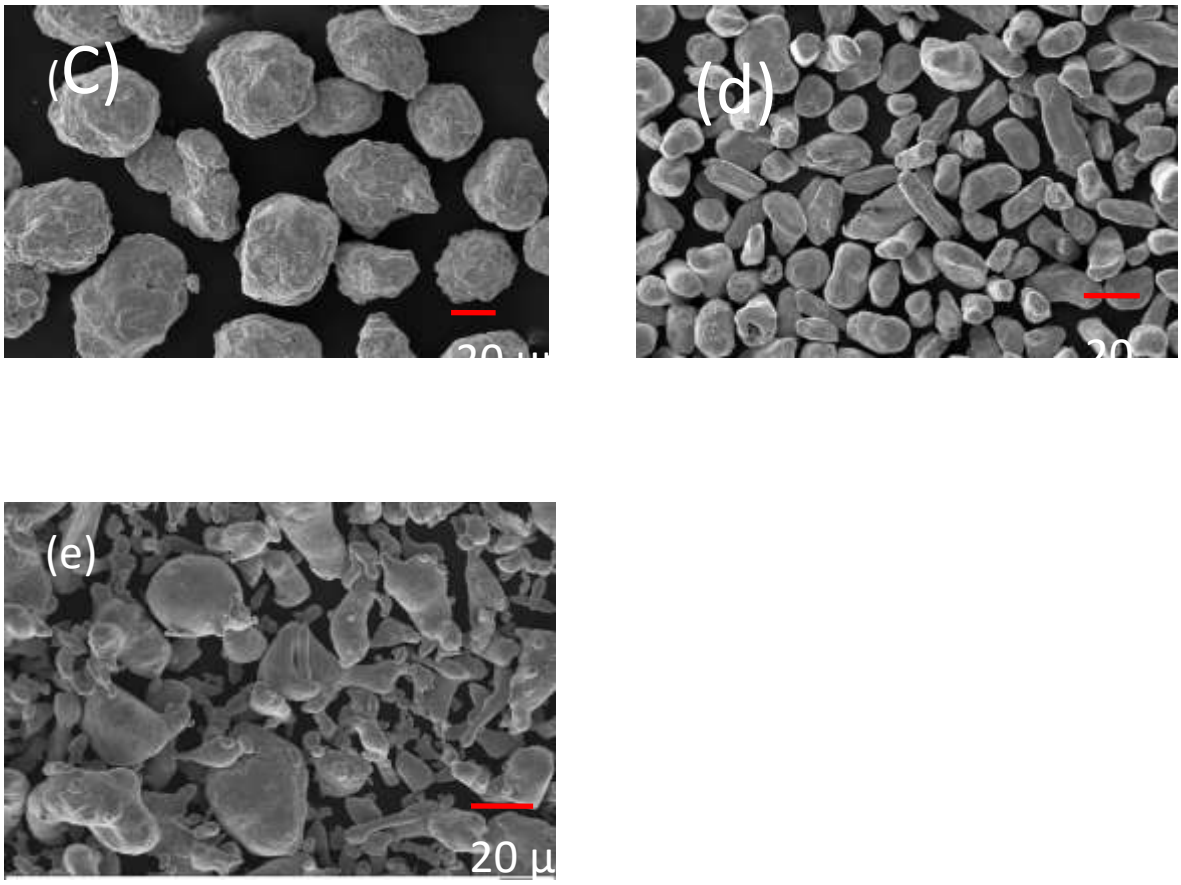
Fig. 2 (a)-(e) presents the scanning electron microscope images of the pure Al, Cr, Ni, Fe, and Zn powders. All powders displayed irregular shapes, with particle sizes approximately 40 µm, as confirmed by the SEM analysis.

Fig. 3 illustrates the X-ray diffraction patterns of the AlCrNiFeZn high entropy powders after various milling durations (0 h, 5 h, 10 h, 20 h, and 30 h). The presence of BCC structures was attributed to chromium and iron, while FCC structures were associated with aluminum and nickel. Additionally, a minor amount of hexagonal close-packed (HCP) structures was identified due to the zinc content. With increasing milling time, the X-ray diffraction peaks broadened, and their intensity decreased, indicating structural refinement. These observations align with previous studies [12–14]. After 30 h of MA, resulted powders contains more BCC phases and small FCC phases and HCP phases were diminished due to structural refinement.

Fig. 4(a)-(e) shows the scanning electron microscope images of the HEA powders at different milling times. Up to 10 hours, cold welding was predominant, resulting in particle agglomeration. From 10 to 20 hours, fracturing began to dominate, leading to finer particles. At 30 hours of milling, nearly equiaxed particles were observed, as shown in Fig. 4(e).







Fg.2 Scanning Electron Microscope images of as received pure powders (a) Aluminium, (b) Chromium, (c) Nickel, (d) Iron and (e) Zn

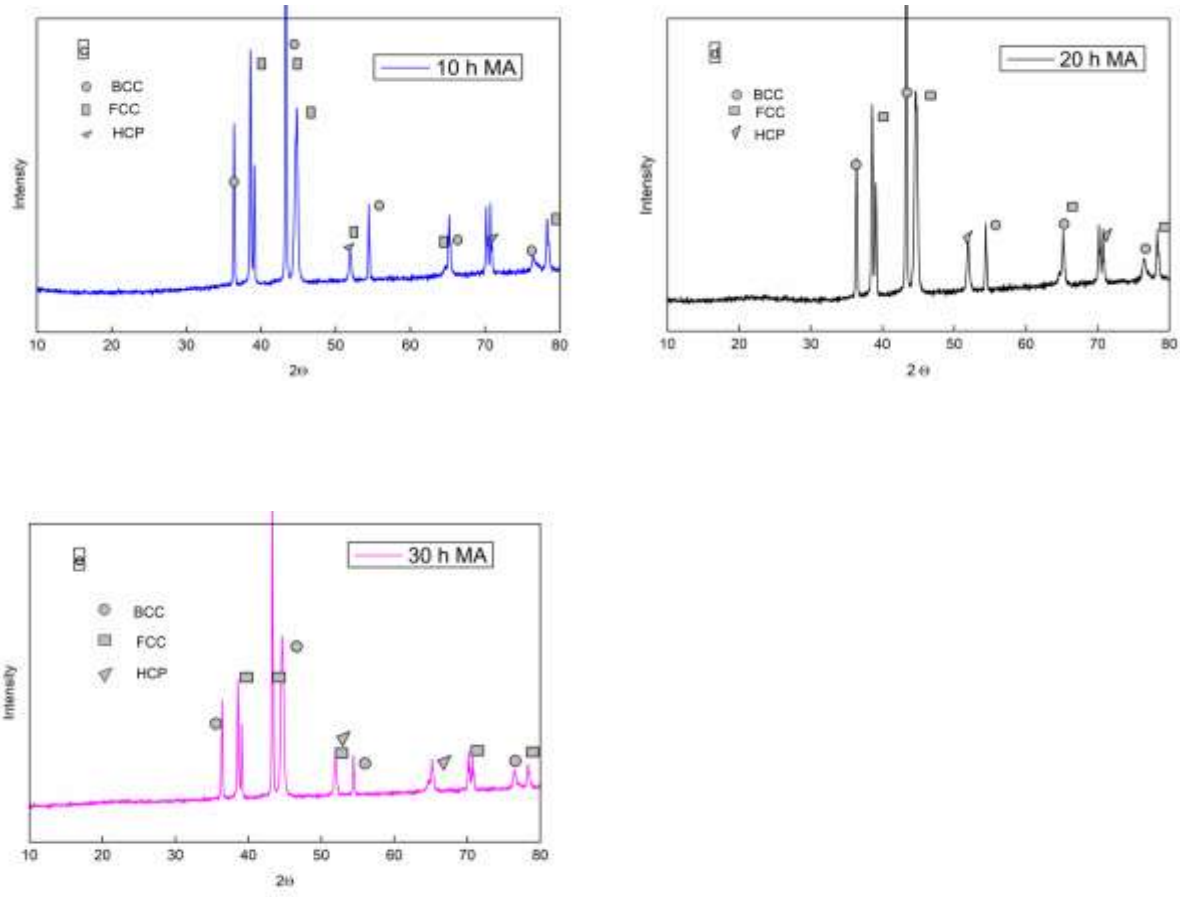


Fig.3 X-ray diffraction images of (a) 0 h, (b) 5 h, (c) 10 h, (d) 20 h and (e) 30 h milled high entropy alloy powders.

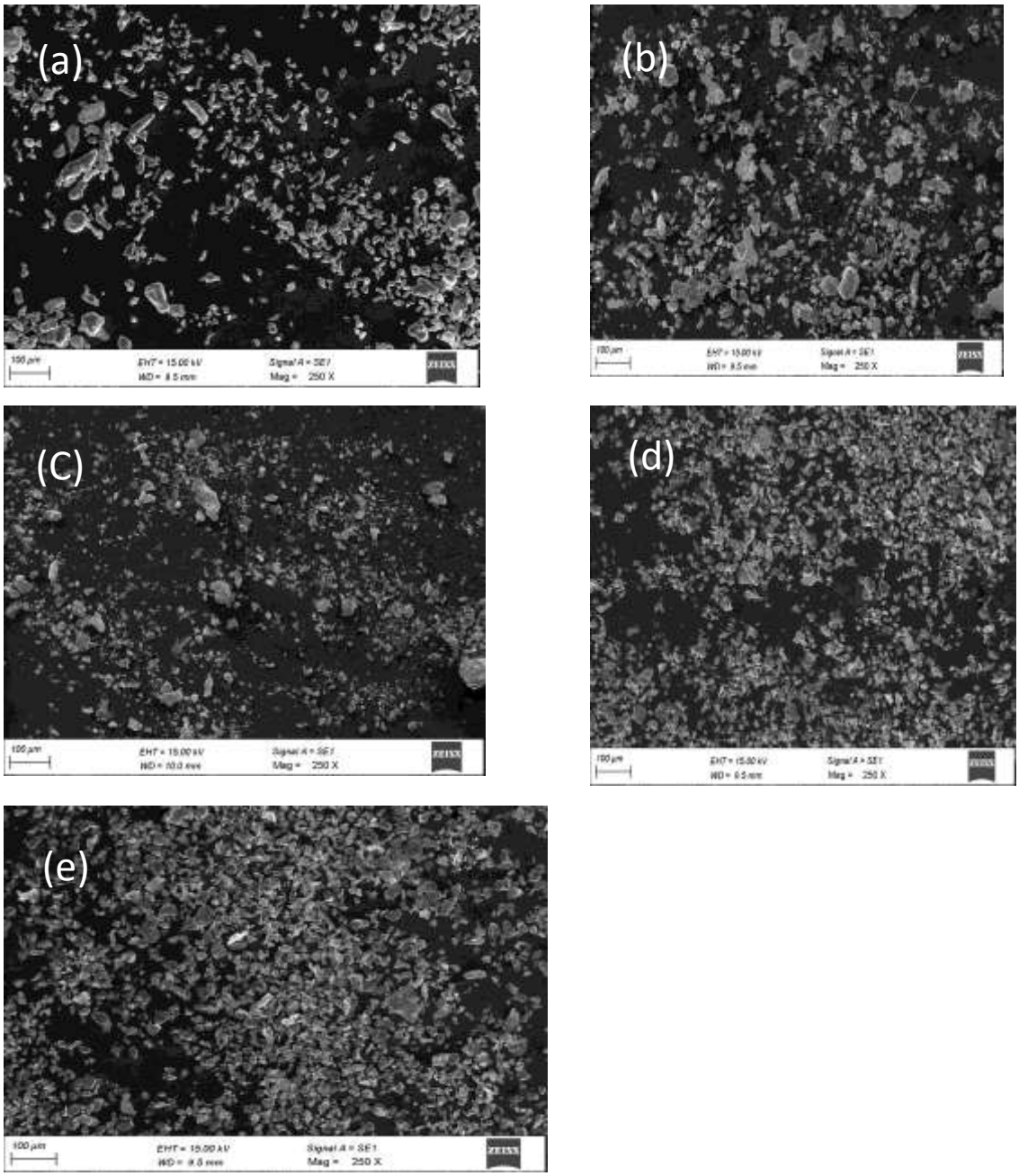


Fig.4. Scanning Electron Microscope images of AlCrNiFeZn high entropy alloy powders after mechanical alloying (a) 0 h, (b) 5 h, (c) 10 h, (d) 20 h and (e) 30 h

Fig. 5 (a) depicts the crystallite size and lattice strain of the High entropy alloy as a function of milling time. Crystallite size, calculated using the Williamson-Hall method, decreased from 150 nm to 45 nm with increasing milling time, while lattice strain increased from 0.05% to 0.65%. This result obtained is in good agreement with previous study [15].

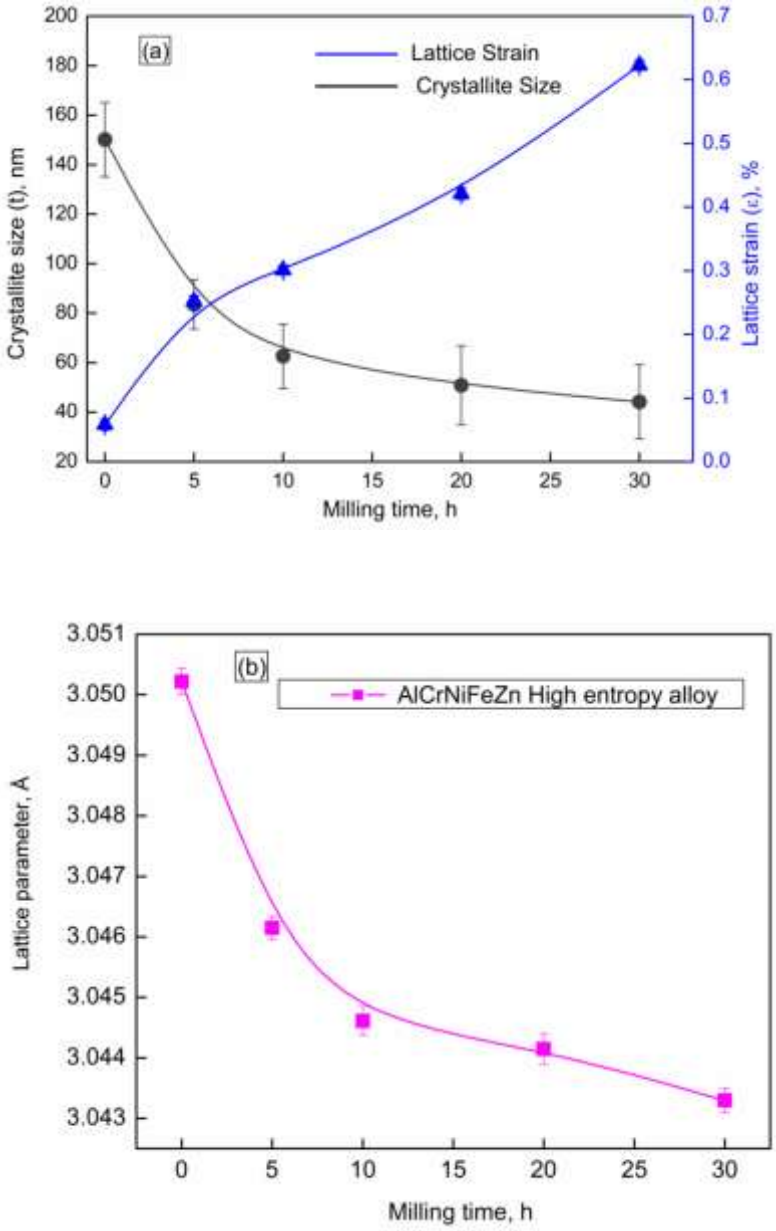
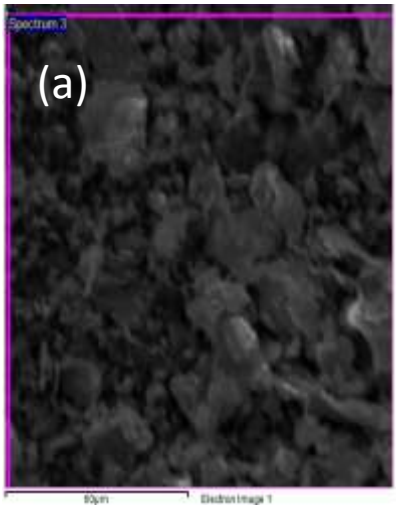
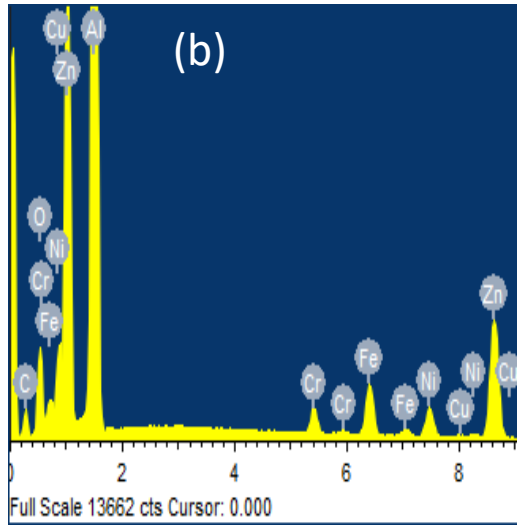


Fig.5. (a.) The crystallite size and lattice strain; (b) The lattice parameter of AlCrNiFeZn high

entropy alloy with various milling time

Fig. 5(b) demonstrates the variation in the lattice parameter of the AlCrNiFeZn high entropy alloy with milling time. The lattice parameter decreased from 3.0502 Å to 3.0435 Å, indicative of densification and structural refinement during mechanical alloying. Fig. 6 shows the energy dispersive spectrum of the AlCrNiFeZn high entropy alloy after 30 hours of milling. The spectrum confirmed the presence of aluminum, chromium, nickel, iron, and zinc in the synthesized alloy powders, validating the composition of the high entropy alloy.





Element	Weight%	Atomic %
C K	13.19	28.78
O K	8.96	14.68
Al K	43.34	42.10
Cr K	1.84	0.78
Fe K	4.38	2.01
Ni K	3.36	1.50
Zn K	24.93	9.99
Totals	100.00	

Fig.6. Energy dispersive spectrum images of AlCrNiFeZn high entropy alloy after 30 h of mechanical alloying

Conclusion

1. AlCrNiFeZn high entropy alloy was successfully synthesized by 30 h of mechanical alloying.
2. Morphological study of prepared high entropy alloy for various milling time was analyzed by scanning electron microscope.
3. After 30 h of mechanical alloying, the crystallite size was 45 nm and the lattice strain was 0.65% was obtained. The lattice parameter value was decreased from 3.0502 Å to 3.0435 Å.
4. Energy diffraction spectrum analysis confirms the element present in the AlCrNiFeZn high entropy alloy.

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