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Characterization of 'AlLiSiTiNi', Equi-atomic High Entropy Alloy Produced by Mechanical Alloying

Ganesh U L[1]
Mechanical Engineering Department, PES Institute of Technology and Management, Shivamogga, Karnataka, India

Raghavendra H[2]
Mechanical Engineering Department, J N N College of Engineering, Shivamogga, Karnataka, India

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***Abstract*—**High entropy based equiatomic (HEA-AlLiSiTiNi) was developed through mechanical alloying and characterized by SEM with EDAX and XRD. The 25 Hrs of ball milling of multicomponent, mechanically alloyed HEA, resulted in Monoclinic phase with better chemical homogeneity, and found to be crystallite size of 30.19 nm with lattice strain induced 0.2897 in as milled condition.

Keywords- High Entropy Alloys (HEA), Mechanical alloying (MA), X-Ray Diffraction (XRD), SEM with EDAX.

1. INTRODUCTION

One or two elements for any component is chosen as main factors and remaining as alloying elements in enhancing the microstructure, properties of the conventional alloy design [1]. Traditionally alloys were manufactured through liquid, solid or gaseous process, during the process the formation of various compounds - intermetallic may leads to changes in properties [2, 3]. However, this conventional approach restricts combination and number of alloy usage, But Yeh et al. in 2004 introduced the concept of HEA's a new way of alloy design and fabrication. It broadening the alloy field and were label at this time as HEAs or multicomponent [4, 5, 6], and we used the above concept in alloy design comprising more than five elements.

HEA's are addressed in two ways - Based on composition or Configurational entropy, per mole.

The present works aims at

- Synthesizing of equiatomic based multicomponent nanocrystalline "AlLiSiTiNi" pentanary, HEA by mechanical alloying through high energy ball milling.
- Microstructure evolution was done through –SEM with EDAX.
- Phase evolution during milling is identified using XRD analysis.

Hence, relying on the literature review, the processing of lightweight HEAs via a simple microstructure, properties and value-effectiveness is technologically important. Thus the present field of research concentrates on an attempt to synthesize HEA by identifying the lightest, structural elements namely Aluminum-Al and Lithium-Li. Families of alloys identified predominantly as

Transition Metal - 3d, Refractory Metal HEAs, Other Alloy Families.

2. EXPERIMENTAL WORK

A. Materials and Chemical Composition of Materials

The characteristics of the 5 pure metal powders Al, Li, Si, Ti, and Ni are shown in TABLE I and the atomic and weight % of these elements are shown in TABLE II.

TABLE I. THE CHARACTERISTICS OF THE PROCURED POWDERS

Sl no	Powder Elements	Particle size	Purity	Melting Point	Density
1	Aluminum	325 Mesh	99.55%	660.3^{0}C	2.7 g/cc
2	Lithium	150 Mesh	99.50%	723^{0}C	0.534 g/cc
3	Titanium	325 Mesh	99.50%	1670^{0}C	4.506 g/cc
4	Nickel	325 Mesh	99.16%	1455^{0}C	8.908 g/cc
5	Silicon	325 Mesh	99.87%	1414^{0}C	2.33 g/cc

TABLE II. CONVERSION FROM AT% TO WT%

Sl no	Powder Elements	Atomic %	Atomic weight	P	W = Chemical compositions in wt%
1	Aluminum - Al	20	26.9815	539.63	16.0006
2	Lithium – Li	20	6.941	138.82	4.1176
3	Titanium – Ti	20	47.867	957.34	28.3970
4	Nickel – Ni	20	58.6934	1173.868	34.8187
5	Silicon - Si	20	28.0855	561.71	16.6611
	Total - 100		P_{Total} = 3371.368	Total Wt% = 100%	

P = 20 atomic % of Aluminium x atomic weight of 'Al'= 20 x atomic weight of 'Al'

= 20 x 26.9815 = 539.63.

W = Chemical compositions in wt% = (P_{Al}/ P_{Total}) x 100% = 16.0006.

The properties of the above mentioned elements are,

- Aluminum (Al): - lightweight, durability, and strength.
- Lithium (Li):- High specific heat, high thermic conductivity, low viscosity, and very low density.
- Titanium (Ti):- High tensile strength even at high temperatures, lightweight, high corrosion resistance, and ability to withstand extreme temperatures.
- Nickel (Ni):- Strength, ductility, and resistance to corrosion and heat
- Silicon (Si):- As alloy integrals to provide more resistance to the Aluminium, Magnesium, Copper and other metals.

After the selection of elements from the periodic table, they have been taken at an equiatomic level and their weight percentage (from TABLE II) has been confirmed by using the following software – developed by David Molhar, shown below in Figure 1.

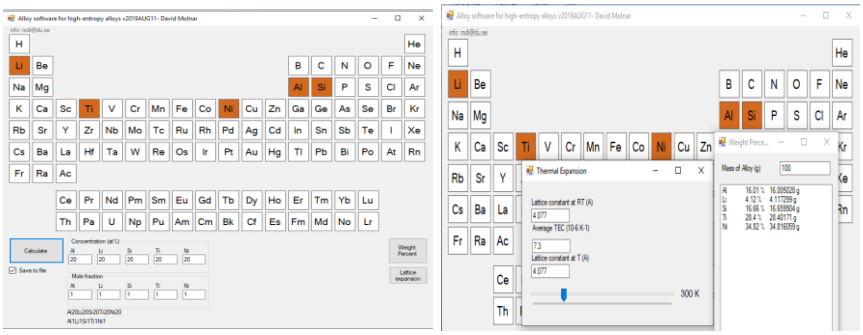


Figure 1. Thermodynamic parameters of the "AlLiTiNiSi" HEA powder

B. Preparation of AlLiTiNiSi HEA Powders by Ball Milling

Mechanical alloying (MA) remains the most preferred technique to prepare solid-state alloys. In a high-energy ball mill, the strategy involves repeated cold welding, splitting (fractured) of powder particles. Physically alloyed blend of 5 pure elements (up to 99% - Al, Li, Ti, Ni and Si) in an equiatomic proportion (20 percent) was shown in Figure 2, using 12 mm diameter PM-100 ball mill, hardened zirconia balls were coated, then sealed in the vial. By keeping a weight ratio of 10:1– ball to powder, the milling was performed at 300 Rpm, up to 25 hours. For every 5 hours that as milled powders were gathered in succession to study the evolution of the process and the sequence of dissolution with respect to the milling period.

Figure 2. PM-100 Planetary Ball Mill

C. Phase Evolution Studies, Scanning Electron Microscopy (SEM), and Energy Dispersive Analysis of X-rays (EDAX)

Phase Evolution Studies - These studies were carried out for identification of phase and crystal structure, for the defined HEA's after mechanical alloying by using the XRD data. The X-ray diffraction studies were carried out in an X'pert Pro (Panalytical, BMSCE-Bangalore) with a Cu-K_α and wavelength of $\lambda = 0.1540598$ nm.

- The XRD system was working at an operating voltage of 40kV and current of 44mA.
- The data acquisition was carried out in a 2θ range of 20°-90° at a step size of 0.02° and scan time was fixed to be 0.7sec respectively.
- The data collected was then analyzed by using X'pert high score plus software and compared to known diffraction files for the phases.
- The XRD works on the principle of Bragg diffraction which is mathematically represented as $n\lambda = 2d\sin\theta$, Where n is the order of reflection, λ is the wavelength of the X-ray, d is the inter-planar spacing of the diffracting planes, and θ is the incident angle of the X-rays.
- The crystallite size was calculated by the Scherrer equation given by,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

- Where D is the crystallite size, K is the shape factor 0.68 to 2.08. K = 0.9 for spherical crystallites with cubic symmetry, λ is the X-Ray wavelength, β the line broadening, and θ is the Bragg angle.

Scanning electron microscope and Energy Dispersive Analysis of X-rays – The as-milled powder material microstructure was characterized with the use of FE-SEM operating at 30 kV, (Nova Nano 450SEM, FEI, BMSCE-Bangalore). The sample was prepared by distributing and transferring the powder on carbon tape and analyzed under SEM. Energy-dispersive X-ray spectroscopy (EDS) detector has described the local phase composition (Bruker, Germany). EDS data were collected from at least 5 points, and averaging the wt % of each item.

Energy Dispersive Analysis of X-rays (EDAX) - when the electron beam interacts with the specimen, EDAX attached to the SEM analyzes the typical X-ray radiation emanated from the specimen. This forms the basis for a chemical composition. The intensities of the peaks can be compared with the peaks in the normal sample in order to determine the relative concentrations of each atomic material.

3. RESULTS AND DISCUSSIONS

A. X-Ray Diffraction (XRD)

The XRD pattern of equimolar HEA (AlLiSiTiNi) powder samples that were sequentially collected at pre- decided interval of time from 5, 10, 15, 20, 25 hrs of ball milled are shown in Figure 3. From the XRD trends it is found that with increased milling hours the amplitude of peaks corresponding to the individual elements decreases drastically. It provides the information on phase evolution of the collected powdered sample. Here as the milling time increases, Al dissolves first, consequently peaks of Al completely disappear after 10 hours of ball milling observed from the peaks of the XRD graph. Later Si, Ti starts to dissolve after 10 hours of ball milling process as indicated by a decrease in the relative intensity of peaks.

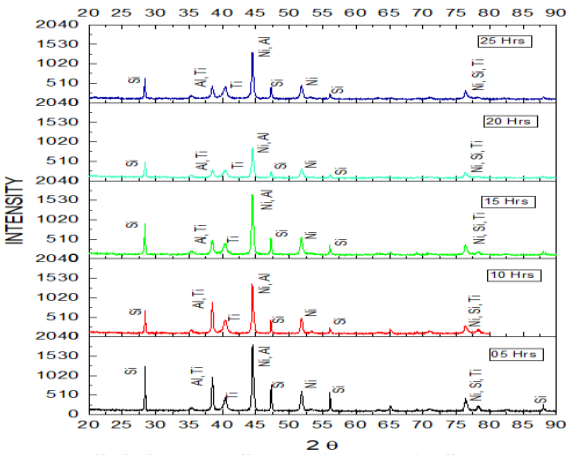


Figure 3. XRD pattern of as milled AlLiSiTiNi alloy as a function of milling time (5, 10, 15, 20, 25Hrs)

The reduction in the intensity of individual peaks and the formation of single peak indicates the formation of single solid solution (C.Sajith babu). After 25 hours of milling, the microstructure of as-milled AlLiSiTiNi powder consists of the Monoclinic phase. The XRD data are tabulated with the help of a high score plus soft- ware and database to match the results shown in Figure 4, and their values are shown in the TABLE III.

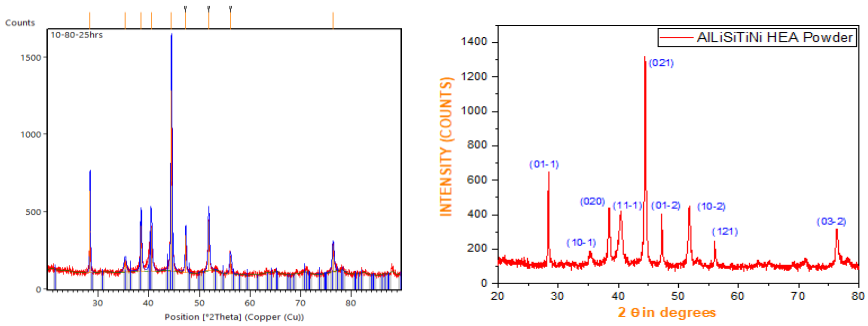


Figure 4. Indexed Peaks for 25Hrs of XRD on AlLiSiTiNi

Crystallite size and Lattice strain –

Crystallite size was estimated by using Debye-Scherrer formula

$$D_P = K\lambda / B \cos\theta$$

Lattice strain of the HEA powders under different milling times are given by

$$\epsilon = \frac{\beta}{4 \tan\theta}$$

TABLE III. CRYSTALLITE SIZE AND MICRO-STRAIN FOR 25 HRS

No.	Pos. [°2Th.]	d- spacing [A]	Height [cts]	FWHM Left [°2Th.]	h	k	l	Crystallite Size only [nm]	MicroStrain only [%]
1	28.39	3.142	541.2	0.147	0	1	-1	55.56	0.226434
2	35.31	2.541	73.3	0.590	1	0	-1	14.13	0.793666
3	38.45	2.340	304.1	0.295	0	2	0	28.52	0.355787
4	40.46	2.229	288.4	0.413	1	1	-1	20.50	0.477
5	44.47	2.037	1191.	0.265	0	2	1	32.33	0.271614
6	47.29	1.922	245.9	0.236	0	1	-2	36.74	0.223546
7	51.82	1.764	327.8	0.295	1	0	-2	29.94	0.255287
8	56.12	1.638	110.7	0.354	1	2	1	25.44	0.281204
9	76.37	1.247	173.7	0.354	0	3	-2	28.56	0.190023

Average Crystallite size – 30.19nm and Average Micro strain – 0.2897

TABLE IV. CRYSTALLITE SIZE AND MICRO STRAIN AT DIFFERENT MILLING TIME

Si. No	Milling Time	Crystallite Size	Micro strain
1	05	44.10	0.2331
2	10	38.88	0.2862
3	15	43.70	0.2575
4	20	31.95	0.2718
5	25	30.19	0.2897

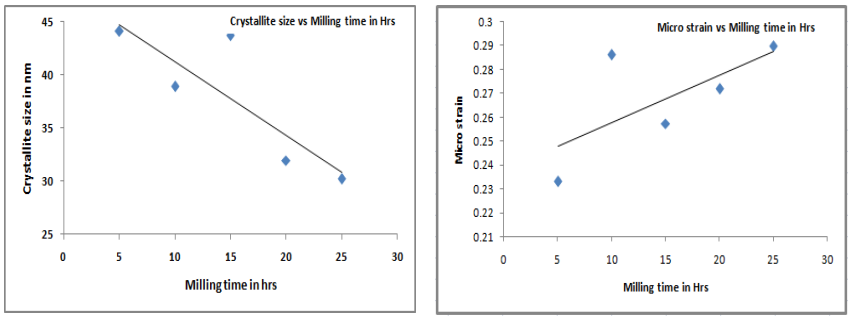


Figure 5. Effect of Milling time in hrs on the Crystallite size and Micro strain

Generally lattice strain increase is due to i) size mismatch between the constituent elements ii) increase grain boundary fraction iii) excessive mechanical deformation. The values of D_p – Crystallite size and ε - Lattice strain with various milling time from 5 to 25 hrs calculated from X'pert high score plus software. From Figure 5, it is observed that as milling prolongs crystallite size decreases and lattice strain increases.

B. SEM with EDAX

Figure 6 displays the AlLiSiTiNi powder SEM photos milled over different time intervals. All of the micrographs are observed with good magnification. Initial as-received powder particle is seen as agglomerated, huge. Yet particle size is slowly decreasing as friction progresses. The structure can be seen to be relatively fine and homogeneous with an improvement in milling time.

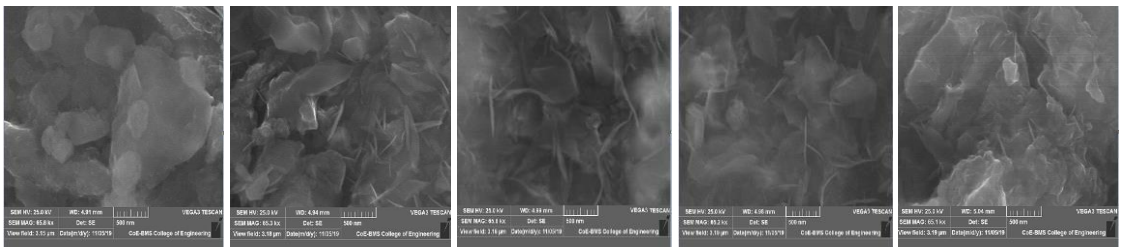
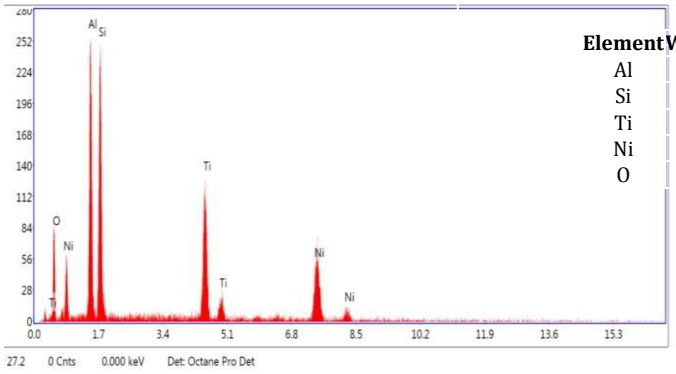


Figure 6. For 05 Hrs, 10 Hrs, 15 Hrs, 20 Hrs, 25Hrs

The experimental spectrum and quantitative analysis of 25 hr milled alloyed by EDS are shown in figure 7. The spectrum was examined by taking from all of the HEA micrograph. The spectrum shows presence of Si, Ni, Al, and Ti. The compositional values are shown in Table 3.3.



Element	Weight%
Al	20.24
Si	20.48
Ti	14.17
Ni	19.37
O	25.74

Figure 7. EDS spectrum and quantitative analysis of AlLiSiTiNi powder milled for 25hrs

TABLE V. THERMODYNAMIC PARAMETERS FOR THE FORMATION OF SOLID SOLUTION

Composition	ΔS_{config}	ΔH_{mix}	VEC	$T_m(K)$	Ω
AlLiSiTiNi	13.3809	-33.76	4.4	1348.24	0.5344

4. CONCLUSION

The synthesis of equiatomic nano-structured AlLiSiTiNi HEA platform was provided by mechanical alloying. After powder milled up to 25 hours, development of nano-structured with a monoclinical crystal is observed. They show the nanocrystalline structure with a crystallite size 30.19nm with a lattice strain of 0.2897%. The homogeneity is confirmed by EDX microanalysis.

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