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The Synthesising Process and Analysing of CoFeNiAlTiCr High Entropy Alloy by Mechanical Alloying

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The high entropy alloys are formed complex structure by synthesis of 4 or more elemental metals and consisted more than 5 atomic percent. The regular phases formed by the alloys occured high entropy. However, the unique properties of the complex structure make these types of alloys special materials. These type of materials show high strength values, especially at high temperatures. Some types of high entropy alloys have high fracture toughness at low temperatures. However, its features are not valid only with high temperatures and low temperatures. In this study, the synthesis of high entropy alloy that contains different elements with different mechanical properties according to Home-rothery rules by mechanical alloying method was performed which has elements different mechanical properties but according to hume-rothery rules. After blending high purity Co, Fe, Ni, Al, Ti, Cr alloys, 1h, 48h, and 96h milling were performed. The obtained mechanical alloy powders were then analyzed by SEM EDS and XRD analyzing methods.

Keywords: High entropy alloy, XRD, copper matrix

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1. Introduction

Thanks to the technology that is constantly and rapidly developing, the materials are diversified and improved. High entropy alloys (HEA), a new material definition, have become a good alternative material group due to their different and high properties[1, 2]. According to recent research, HEAs have been reported to exhibit unique properties such as corrosion and abrasion resistance, high hardness, strength stabilization at high temperatures, high ductility and hardness[1, 3, 4]. This features make the material concept special. HEA is a multicomponent alloy type with an atomic ratio of 5-35%. Thus, the idea of a conventional alloy type was broken[5, 6]. Their structures are generally present in the solid phase of BCC (hard), FCC (soft), BCC + FCC and HCP [7]. The material group that holds both phases together gains the properties of the phases it holds. This type of phase, where the same material is hard and ductile, may appear HEA[1, 3, 6]. Due to the placement of the microstructure of this type of material group which consists of 5 and more components in different proportions, the configuration entropy is high. HEA can be synthesized by many methods [8]. In these methods, arc melting furnace and induction melting furnace with fast heating speeds can be used. They can also be used in high-energy ball mills. In this study, CoFeNiAlTiCr HEA alloy was synthesized using a ball mill and characterization analyses were performed.

2. Experimental details

In this study, CoFeNiAlTiCr, which is HEA, was synthesized using mechanical alloying method. High purity Co, Fe, Ni, Al, Ti, Cr elemental metals molar ratios Co, Fe, Ni, Al0.4, Ti0.6, Cr0.5 were weighed. Then, zinc stearate was added to prevent the samples from overheating and plastering. These processes were carried out in argon environment to prevent oxidation of the materials during alloying. In the studies, mechanical alloying has been made by using high-energy ball mill (Figure 1.).

***Fig.1**: Ball milling device.*

The weighed powder mixture was adjusted to a ball-to-powder ratio of 15: 1 and placed into the container using 12mm diameter steel balls. The powder mixture was prepared with the Retsch PM 100 instrument at 250rpm at different times. The obtained samples were prepared for DTA, SEM, XRD and EDS analyzes and the results were taken.

3. Results and discussion:

The samples were applied grinding process during 1hour, 48 hours and 96 hours, respectively. The differential thermal analysis (DTA) [9-11] measurements were taken by using a Shimadzu TA-60 WS. The all plots are combined in single plot and it was presented in Figure 2.

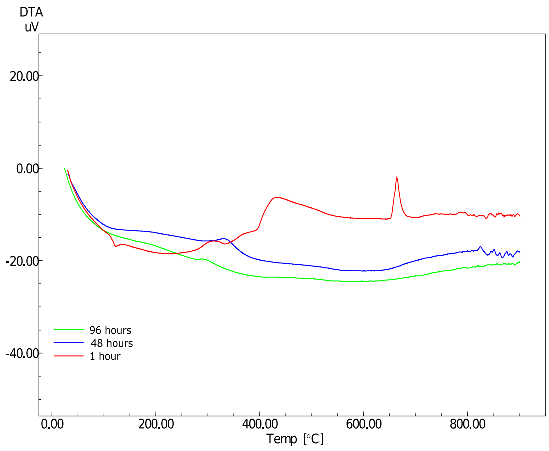


***Fig.2**: Scale and ball milling device*

During grinding, due to the character of the grinding process, cold welds and breakage of the powders occur again. At this time, grinding agents were used to prevent agglomerates and to maintain the grinding process. These agents left the structure before reaching 200 degrees, as shown in the DTA analysis in figure 2. The reason why this occurs only in the 1 hour sample can be explained as follows; with increasing grinding time, the temperature rise in the local areas of the dust can reach up to 200 degrees in places. This indicates that the grinding agents move away from the structure during grinding. With an increasing temperature after 200 degrees, an exothermic reaction [9, 11] appears to be about 600 degrees per hour in the sample. Grinding for one hour does not cause any change in the powders, but only allows them to mix homogeneously. Exothermic reaction in one hour sample shows that an intermetallic compound is formed by the effect of temperature between two elemental powders in the oil. This suggests that the intermetallic compound may be an aluminite in the form of FeAl or NiAl. The absence of such a reaction in the DTA analysis of the milled samples for 48 hours and 96 hours indicates that no intermetallic compound is formed and that the samples convert to a stable structure during milling. It is clear that this structure is HEA. If HEA had not been formed during milling, an intermediate phase formation would necessarily occur with increasing temperature at the end of milling.

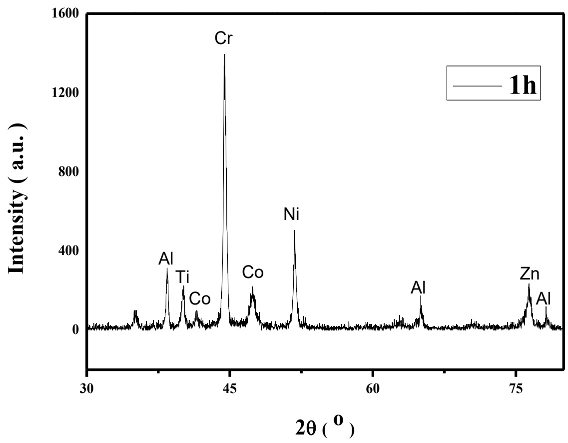


***Fig.3:** XRD pattern for 1 h.*

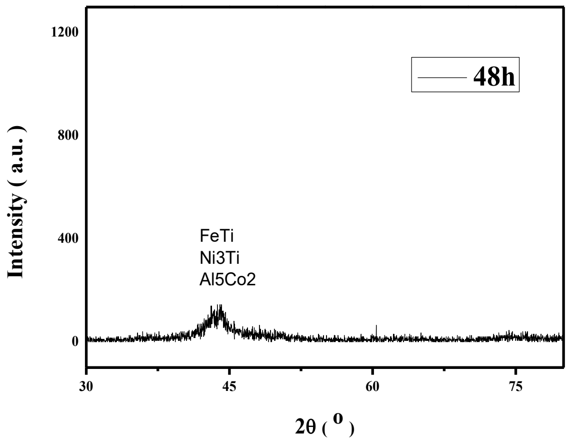


Fig.4: *XRD pattern for 48 h.*

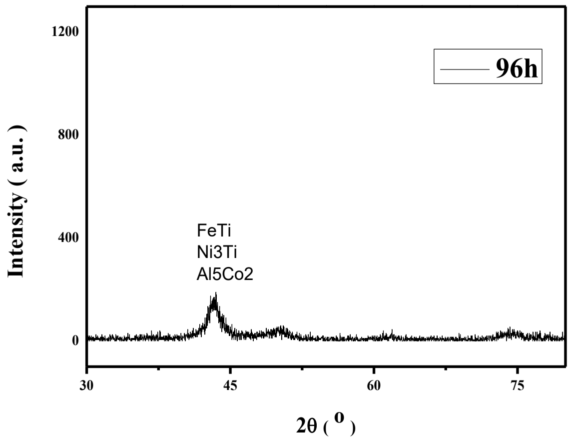
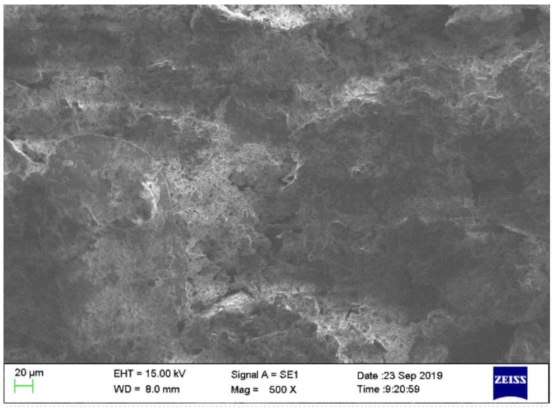


Fig.5: *XRD pattern for 96 h.*

In this study, the reason for sample after one hour of grinding is to show that the powders are homogeneously mixed and the powder mixtures given in the experimental part are used. This is clearly seen in the XRD analysis given in Figure 3. At the end of one hour grinding process, Co, Fe, Ni, Al, Ti, Cr peaks in the elemental state in the structure are clearly seen. With the increase in grinding time, the crystalline structures of all powders, which had mechanical alloying, began to degrade and disintegrate due to ball impacts, and complete amorphization occurred in the later stages of the grinding. At 48 hours of milling, a large peak of about 43 degrees is observed where dust recrystallization occurs again.

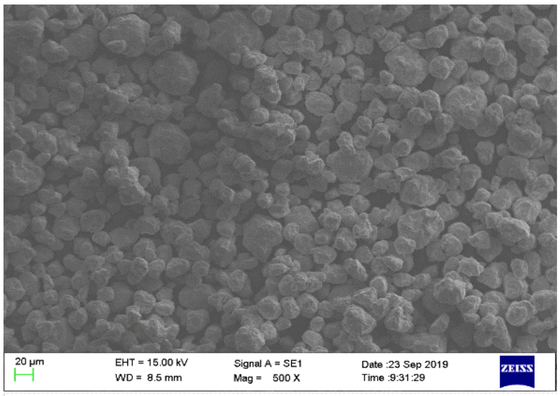


Spectrum: Object 2

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Co	27	K-series	6360	62.97	46.46	42.84	2.12
Ni	28	K-series	2626	30.55	22.54	20.87	1.20
Ti	22	K-series	4271	15.07	11.12	12.62	0.54
Fe	26	K-series	1945	13.73	10.13	9.85	0.57
Cr	24	K-series	1830	8.13	6.00	6.27	0.35
Al	13	K-series	2023	5.08	3.75	7.54	0.31
			Total:	135.52	100.00	100.00	

Fig.6: *SEM images and EDS results for 1h.*

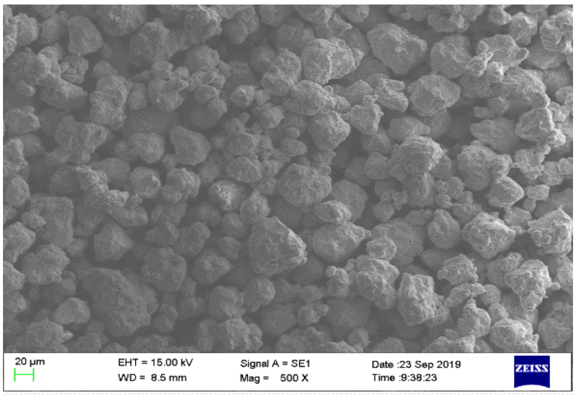
Although it shows that there is a small amount of crystallized structure in this peak structure, there are still high amorphous structures. With the milling going out for 96 hours, the intensity of the peak at 43 degrees in figure 5 increased further.



Spectrum: Object 2

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Co	27	K-series	3329	30.35	26.56	24.53	1.12
Fe	26	K-series	4366	29.28	25.62	24.97	1.03
Ni	28	K-series	2424	26.26	22.98	21.31	1.05
Ti	22	K-series	4288	13.80	12.08	13.73	0.49
Cr	24	K-series	2924	12.08	10.58	11.07	0.46
Al	13	K-series	1089	2.48	2.17	4.38	0.18
			Total:	114.26	100.00	100.00	

Fig.7: *SEM images and EDS results for 48 h.*



Spectrum: Object 2

El	AN	Series	Net	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Co	27	K-series	1325	29.57	26.31	24.36	1.32
Fe	26	K-series	1785	29.28	26.05	25.46	1.20
Ni	28	K-series	986	26.14	23.26	21.62	1.28
Ti	22	K-series	1714	13.50	12.01	13.69	0.57
Cr	24	K-series	1161	11.73	10.44	10.96	0.54
Al	13	K-series	388	2.17	1.93	3.91	0.20
			Total:	112.40	100.00	100.00	

***Fig.8**: SEM images and EDS results for 96 h.*

This has been shown to increase crystallization with increasing grinding time. In addition, it can be said that new peaks are formed at 48 and 70 degrees. With the etching of the powder obtained after 96 hours, all of the amorphous phases in the structure can be removed from the sample and the crystallized HEA structures remain.

SEM and EDS results are given in Figure 6-8. With increasing grinding time, it can be said that the diameter of the powder particles exceeds 20 microns. The reason for this situation can be explained as follows. Powders whose particle sizes decrease with increasing grinding time tend to agglomerate. Although the grinding agent contained in the structure prevented this situation, the amount of flocculation in the powders increased with the removal of the agents from the structure after 48 and 96 hours of grinding. These pellets with a particle size above 20 microns contain both HEAs and amorphous powder mixtures. Given EDS analysis, with the increased grinding time, Co, Fe, Ni, Ti, Cr, Al elements on all sides of the powder particles are close to the planned ratio at the beginning of the desired HEA.

Conclusions

In this study we have focused on synthesizing high entropy alloys which is consisted Co, Fe, Ni, Al, Ti, Cr. These elements were prepared regarding molar ratio and alloyed with mechanical alloying milling. The obtained alloys analyzed with different device. The XRD peaks have shown us the over 48h process the high entropy alloy was occurred and SEM provided particle images. It was evident that at the end of the 1 hour milling process no change in the powders occurred. It was seen that HEAs started to form with increasing grinding time and their amount increased after 96 grinding. It can be clearly stated that HEA production in mechanical alloying is successful.

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