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Influence of Sintering Temperature on Microhardness and Tribological Properties of Equi-Atomic Ti-Al- Mo-Si-W Multicomponent Alloy

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Abstract. This work aim to investigate the effect of sintering temperature on microhardness and tribological properties of novel equi-atomic TiAlMoSiW HEA fabricated via spark plasma sintering. The influence of Spark plasma sintering temperature on morphological evolution and phase formation was also investigated. The microstructure and the phases formed for the developed HEA were examined using scanning electron microscopy (SEM) and X-ray diffractometry (XRD) respectively. The microhardness and tribological properties were studied using a diamond base microhardness tester Rtec tribometer. It was noticed that sintering temperature has an effect on microhardness and tribological properties. Phase analysis of the samples displayed that the alloy exhibited a bcc matrix with secondary phase precipitate of ordered fcc TiSi_2 phase. The developed HEA showed improved mechanical properties as the sintering temperature increases.

1. Introduction

Due to industrial development, advanced engineering materials with good strength to weight ratio and wear resistant materials are needed [1]. HEAs are promising multi principal element alloys with good mechanical properties for advanced engineering application [2-5]. Today, HEAs remain the promising multi principal element alloys with combination of outstanding chemical and mechanical properties for advanced engineering application at both room and elevated temperatures [6-8]. Numerous methods are followed by many authors in fabricating these class of alloys. Other authors used techniques such as spark plasma sintering, sputtering, additive manufacturing (LAM) and casting until forming hard and advanced coatings using laser cladding techniques and others [7]. However, in these work spark plasma sintering (SPS) is used as a fabrication technique. SPS has an ability to consolidate a material which possess good mechanical properties. These is achieved by controlling the grains size of the developed alloy as compared to other conventional methods [9, 10].

Chuang et al, [11] investigated the wear resistant of $\text{Co}_{1.5}\text{CrFeNi}_{1.5}\text{Ti}$ and $\text{Al}_{0.2}\text{Co}_{1.5}\text{CrFeNi}_{1.5}\text{Ti}$ high entropy alloys. Both alloys showed outstanding wear properties at room temperature. The developed alloys showed better wear properties as compared to conventional wear resistant steels. In many investigated tribological studies, abrasive conditions and adhesive conditions are used to conduct tests for wear behaviour of HEAs. Wu et al. [8] studied the effect of alloying composition ratio on wear behaviour of $\text{Al}_x\text{CoCrCuFeNi}$ HEAs. The author discovered that higher Al content in the fabricated



alloy results in worn surface been smooth and it also yields fine debris with a high oxygen content, which also results in a large improvement in wear resistance of the developed alloy.

Therefore, in this study, an attempt was made to fabricate Ti-Al-Mo-Si-W HEAs under different sintering temperature. Therefore, in depth characterization of the synthesized HEAs was undertaken to understand the effect of sintering temperature on microhardness and tribological properties, microstructures and formation of phases of the developed HEAs.

2. Methodology

Ti-Al-Mo-Si-W equi atomic HEA powders were mixed mixer and poured into a graphite die of 40mm diameter, the powder were sintered using an SPS system (FCT Systeme GmbH Rauenstein, Gewerbepark 96528 Frankenblick). Temperatures of 800, 900 and 1000°C at a heating rate of 100°C/min with a holding time of 8min were used as sintering parameters. A constant pressure of 50 MPa was applied during the sintering process. The fabricated HEAs samples were cut transversely, mounted in an epoxy- based resin, polished using metallography procedures and etched with kroll agent solution for investigating the microstructural evolution of the developed alloys. The microstructures of sintered and worn Ti-Al-Mo-Si-W was characterized by means of SEM. The phases present in the synthesized high entropy alloys were identified using an x-ray diffractometer and present phases were revealed using X-Pert High Score Plus software. Vickers microhardness (HVN) measurements of the fabricated samples was obtained using an Emco TEST microhardness tester. The indenting load of 100kgf and a dwell time of 10s was used. The fabricated HEAs samples surfaces were indented randomly five different positions and the average was recorded. Tribological studies were examined using Rtech tribometer with a stainless steel contact ball. The applied force was 150N for a duration of 5min.

3. Results and Discussion

Table 1 present the pattern used to sintered the multicomponent HEAs

Table 1. Summarized pattern of fabricated TiAlMoSiW HEAs.

Alloy	Temperature (°C)	Pressure (kPa)	Holding Time (min)	Heating Rate (°C/min)
Ti-Al-Mo-Si-W	[1A] 800	50	8	100
	[1B] 900			
	[1C] 1000			

SEM and XRD Analysis

SEM micrographs of the fabricated Ti-Al-Mo-Si-W high entropy alloy at 800 °C, 900 °C and 1000 °C were taken and recorded in Figure 1. The reported set of samples were sintered at 50MPa pressure and sintering holding time of 8 min each. For each sintering temperature effect, the SEM results of the HEAs surface has no pores or crack for the samples sintered at all temperatures. However, the micrograph revels minimal cavities were 1000 °C has more cavities as compared to samples sintered at 800°C. Due to low melting point of Al, the cavities could be due to the burning off Al. The alloys display morphology of different phases with gray, white and small black precipitated spots contrast phases present. From the three distinctive areas are visible in all micrographs present in Figure 1a-c, which are irregular bulk areas of gray, white phases and irregular black hole areas can be observed. The gray and white phases are possibly the main phase in the alloy due to the fact that they account for a higher volume fraction on the surface.

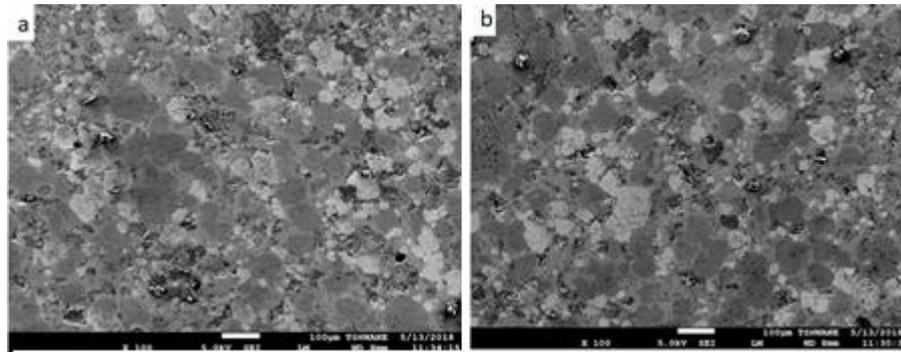


Figure 1. SEM image of the sintered HEA a) 800oC and b) 1000oC

Table 2. The calculated parameters (ΔS_{mix} ($\text{JK}^{-1}\text{mol}^{-1}$), ΔH_{mix} (KJ/mol), T_m (K) δ (%) and Ω for the investigated alloys.

Alloy	ΔH_{mix}	ΔS_{mix}	T_m	δ & Ω
Ti-Al-Mo-Si-W	-24.32	13.38	2230	7.97 & 1.22

Table 2 presents the empirical values of mixing entropy and enthalpy of Ti-Al-Mo-Si-W high entropy alloy. It is anticipated that HEAs forms a solid solution phase (BCC/FCC) when the defined parameters are $\Omega \geq 1.1$ and $\delta (\%) \leq 6.6\%$ [13-15]. Therefore, from the calculated results, the defined parameters of Ω and δ (%) for the TiAlSiMoW high entropy alloy system was found to be 1.22 and 7.79 % respectively. It is with no doubt that Ω parameter matches the proposed value while $\delta(\%)$ defined parameter breaks the simple crystalline structure formation rules for the multi-principle HEAs proposed by Zhang et al. [15]. Intermetallics or complex phases are expected to be found in the alloy system as confirmed by the XRD results with intermetallic of Titanium silicide present in the alloy from figure 2 below. Figure 2 present the phases present in the developed Ti-Al-Mo-Si-W sintered at 800, 900 and 1000°C. The XRD shows that the bulk Ti-Al-Mo-Si-W HEAs consists of combination of BCC and FCC phase with the present of TiSi_2 . The combination of BCC and FCC in the alloy is expected to give better mechanical properties. The developed compound was due to high entropy of mixing of the binary components (Ti, Si). The compound is also known for superior properties such as high strength, high melting point, low density, outstanding oxidation resistance and these properties gives the compound a potential aero-space engineering material [10].The combination of both BCC and FCC in the alloy system is expected to give better mechanical properties.

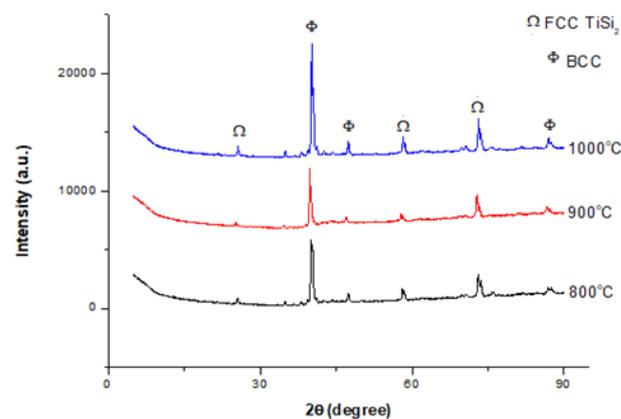


Figure 2. XRD patterns of sintered Ti-Al-Mo-Si-W high entropy alloys

Microhardness and CoF Results

Figure 3 displays the microhardness and CoF properties of the developed high entropy alloy of Ti-Al-Mo-Si-W sintered at different temperatures. The relationship between microhardness and CoF can not be ignored. It is evident that as the sintering temperature increases, the microhardness of the fabricated Ti-Al-Mo-Si-W HEA increases while the CoF decreases. An increase in microhardness values is ascribed to particle to particle rearrangement and uniform distribution of present phases in the alloy [15]. These homogenous distribution of secondary phase precipitates on the grain boundaries which tends to restricts the grains growth and hence it impedes dislocations motion leading to improved microhardness values. The obtained results are inline with Makena et al. [16], Otto et al. [17] and Shongwe et al.[18]. Maximum microhardness of 802.93 HV_{0.1} was evident at sintering temperature of 1000°C with minimum CoF of 0.0916μ.

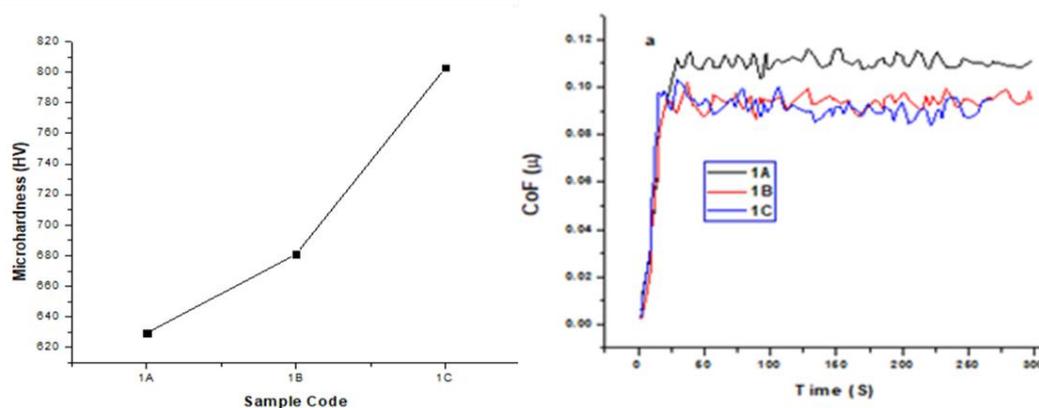


Figure 3. Microhardness and CoF results of the sintered HEA

SEM Analysis of the Worn Surface

The SEM images in figure 4 illustrate the effect of sintering temperature on the wear properties of the developed equi-atomic TiAlMoSiW HEAs. The SEM image shows material carry overs of the HEAs due to minimal plastic deformation occurs in the direction of the sliding movement. For a high load of 150N from the contact ball, the developed HEAs exhibit good wear resistance as there are no defaults on the surface. Good microhardness properties of the synthesized HEAs could be responsible for the outstanding wear properties. Also the combination of both BCC and FCC in the alloy system which was found in the Xrd results proved to have better mechanical properties. All the fabricated HEAs resulted in a smooth surface which displays no plastic deformation by the contact ball. The high density, high microhardness and high amount of W could be the reason for minimal wear loss with no evident of furrows tracks as a result of plastic deformation. Only material cracks be detected in specimen 4a and 4c. The SEM image at all sintering temperatures also displays minimal cracks on the surface after wear. The surface shows no degree of deformation which resulted to outstanding wear resistance properties.

Conclusion

TiAlMoSiW HEAs were successfully consolidated via spark plasma sintering and the effect of sintering temperature on the microstructural evolution, microhardness and tribology properties of the synthesized HEAs were investigated. Generally, the synthesized HEAs showed outstanding mechanical properties.

- Maximum microhardness of 802.09HV was achieved at a sintering temperature of 1000°C.
- SEM of the worn samples presented stable surface with minimal deformation

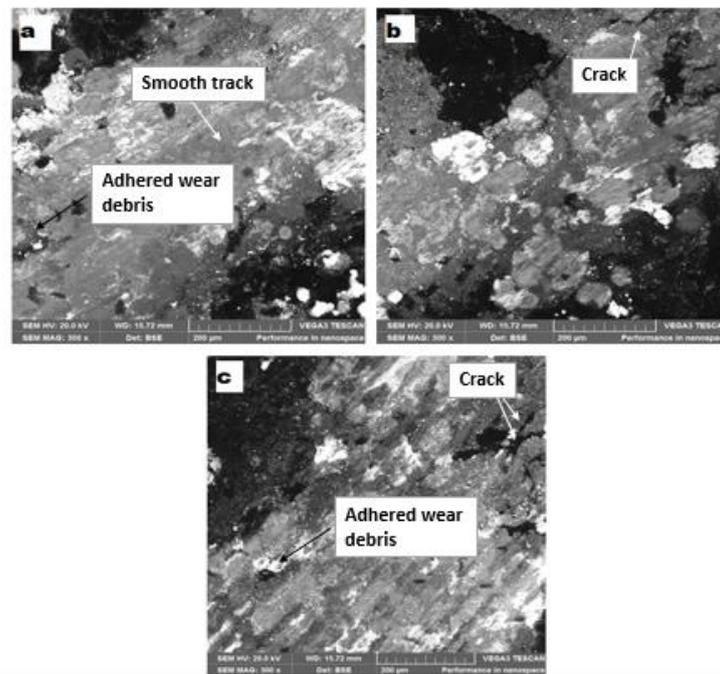


Figure 4. SEM micrographs of wear tested Equi-Atomic TiAlMoSiW high entropy alloy at a) 800 °C
b) 900°C c) 1000°C

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