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Hadi Bilir
Kastamonu University, hadibilir@gmail.com, Kastamonu-Türkiye
Mehmet Akkaş
Kahramanmaraş İstiklal University, mehmet.akkas@istiklal.edu.tr, Kahramanmaraş-Türkiye
Can Doğan Vurdu
Kastamonu University, cvurdu@kastamonu.edu.tr, Kastamonu-Türkiye

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ORCID ID	0000-0002-6293-4138	0000-0002-0359-4743	0000-0002-5179-1266
Corresponding Author	Mehmet Akkaş		

POWDER METALLURGY-BASED PRODUCTION OF HIGH-ENTROPY ALLOYS: STRUCTURAL AND FUNCTIONAL EFFECTS OF SI ADDITION

ABSTRACT

In this study, a CrMnFeCoNi-based high-entropy alloy (Cantor alloy) was modified by the addition of 5 wt.% silicon (Si) to develop a composite structure with improved microstructural and mechanical properties. Si was selected based on its potential to promote phase stability and reinforcement, as well as its frequent use in similar studies. Elemental powders were homogenized via high-energy ball milling to ensure uniform mixing and enhanced atomic-scale dispersion. The mechanically alloyed powders were compacted through uniaxial cold pressing at 600 MPa to increase green density and interparticle contact. Sintering was conducted at 1000 °C in a controlled atmosphere to activate solid-state diffusion and facilitate phase formation and densification. Microstructural characterization was performed using scanning electron microscopy (SEM). The analyses revealed that Si addition influenced the distribution of the reinforcement phase, porosity, and particle morphology. Notably, phase evolution, pore formation, and particle dispersion were strongly affected by both the presence of Si and the applied sintering conditions. The results indicate that 5 wt.% Si significantly enhances microstructural integrity and contributes positively to the overall mechanical performance of the composite material.

Keywords: High-Entropy Alloys (HEAs), Powder Metallurgy, Silicon Addition, Microstructure, Mechanical Properties

1. INTRODUCTION

High-entropy alloys (HEAs), a novel class of metallic materials, have garnered considerable attention in recent years due to their unconventional compositional design and outstanding properties. Unlike traditional alloys that are based on one or two principal elements, HEAs consist of at least five major elements in near-equiatomic proportions [1]. This unique compositional strategy yields high configurational entropy, which promotes the formation of simple solid solution phases—typically face-centered cubic (FCC) or body-centered cubic (BCC)—while suppressing the formation of brittle intermetallic compounds. Among the various HEAs studied to date, the equiatomic CrMnFeCoNi alloy, commonly referred to as the Cantor alloy, stands out due to its exceptional mechanical behavior and microstructural stability [2]. It exhibits a stable single-phase FCC structure with excellent ductility even at cryogenic temperatures, as well as notable hardness, wear resistance, and resistance to corrosion and oxidation [3, 4, and 6]. These properties

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make it a promising candidate for demanding applications in sectors such as aerospace, energy, and cryogenics [7]. Despite the favorable characteristics of HEAs, their properties are highly sensitive to processing parameters and fabrication methods. Powder metallurgy has emerged as an effective technique for HEA synthesis due to its capability to achieve homogeneous elemental distribution, refined microstructures, and controlled porosity. In particular, mechanical alloying followed by cold pressing and sintering is widely used to produce HEAs with enhanced structural and functional properties [8, 9, 10, 11, 12 and 13]. Among the various process parameters, sintering temperature plays a crucial role in determining final microstructural features such as phase formation, grain size, densification, and porosity, which in turn influence the alloy's overall performance [14].

The optimization of sintering parameters is therefore essential to unlock the full potential of HEAs. Variations in sintering temperature can lead to significant differences in phase stability, defect density, and elemental distribution. Previous studies on FeCoCrNi-based HEAs have demonstrated that the choice of sintering temperature directly affects mechanical strength, hardness, and microstructural integrity [13, 14 and 15]. In this work, a CrMnFeCoNi-based HEA was modified by the addition of selected alloying elements and synthesized via powder metallurgy. The primary objective of the study is to systematically investigate the influence of sintering temperature on the microstructural evolution and stability of the resulting alloy system. By evaluating phase formation, porosity, and microstructural homogeneity, the study aims to identify optimal processing parameters that enable the fabrication of HEAs with superior mechanical and functional properties.

2. RESEARCH SIGNIFICANCE

This study provides a novel insight into the development of silicon-modified high-entropy alloys (HEAs) produced by powder metallurgy techniques. By incorporating 5 wt.% Si into a CrMnFeCoNi-based Cantor alloy, the research explores how such an addition influences microstructural integrity and mechanical behavior. The study bridges a significant knowledge gap in the design of HEA-based composite systems, particularly regarding the role of Si in enhancing phase stability and controlling porosity. The use of high-energy ball milling, followed by cold pressing and sintering, offers a scalable and cost-effective route for HEA composite fabrication. The findings contribute to the broader field of advanced structural materials and provide a basis for tailoring HEA properties for targeted engineering applications.

Highlights:

- A CrMnFeCoNi-based high-entropy alloy was modified with 5 wt.% silicon via powder metallurgy.
- Sintering at 1000 °C promoted densification and microstructural refinement.
- Enhanced microstructural integrity and mechanical performance were achieved through Si incorporation.

3. MATERIALS AND METHODS

In this study, the equiatomic CrMnFeCoNi five-element system, commonly referred to in the literature as Cantor alloy, was selected as the base alloy matrix (hereafter designated as the "base component" or BC). Each constituent element was incorporated at approximately equal atomic percentages, precisely 20 at. % each, to maintain the characteristic high-entropy configuration. To enhance the composite properties, silicon (Si) was introduced as a reinforcement element at an atomic concentration of 5%. The sintering temperature was fixed at

1000 °C throughout the sample fabrication process to ensure consistent thermal treatment conditions. The powder mixtures were synthesized using a high-energy mechanical alloying technique aimed at achieving a homogeneous distribution and effective alloying of the constituent elements. The milling was conducted at a rotational speed of 400 revolutions per minute (rpm), a parameter selected to balance the kinetics of alloying and minimize excessive heat generation or particle agglomeration.

Subsequent to the milling process, the powders were compacted into cylindrical specimens with dimensions of 13 mm in diameter and 5 mm in height through cold uniaxial pressing under a pressure of 600 MPa. However, due to the structural characteristics imparted by the silicon reinforcement phase, the applied compaction pressure was reduced in certain samples to avoid potential cracking or excessive deformation. To promote enhanced mixing uniformity and facilitate the penetration of the reinforcing silicon phase into the base matrix, steel milling balls of varying diameters—6 mm, 8 mm, and 10 mm—were utilized concurrently. The quantity and size distribution of the milling media were carefully balanced according to the volumetric capacity of the milling vial, ensuring optimal collision energy and mixing efficiency. The ball-to-powder weight ratio was maintained precisely at 10:1, a ratio determined to be effective in prior optimization studies.

Prior to milling, the tare weights of the milling vial (2387 g) and its lid (772 g) were accurately measured using a high-precision analytical balance to guarantee precise control over the powder mass and thereby maintain the correct ball-to-powder ratio. Additionally, to prevent adherence of powder particles to the vial walls due to frictional heating and high rotational speed, a slight excess of powder was added at the start of milling while maintaining the overall proportionality of components. A detailed overview of the milling equipment, process parameters, and the resulting powder characteristics are summarized in the accompanying tables and equipment specifications section. Following the sintering procedure, the samples underwent metallographic preparation to facilitate microstructural characterization. Initially, coarse grinding was performed using 75-grit abrasive papers to remove surface irregularities and flatten the specimens. Subsequently, a sequence of progressively finer silicon carbide (SiC) papers with grit sizes of 320, 600, 800, 1200, and finally 1500 was employed to obtain a smooth and scratch-free surface suitable for high-resolution microscopy. Each grinding stage was meticulously applied for approximately 3 to 4 minutes, ensuring uniform material removal and surface finish across all samples.

The final polishing step involved the application of a 1-micron diamond suspension, water-based, which was gently applied using a microfiber cloth. This polishing was conducted for no less than five minutes to achieve an optically reflective surface free from deformation-induced artifacts, thereby enabling detailed and accurate microstructural analysis.

4. FINDINGS AND DISCUSSIONS

The morphological and microstructural characteristics of specimens produced via powder metallurgy with silicon reinforcement were systematically investigated using scanning electron microscopy (SEM) analysis (Figure 1). The data obtained from these SEM examinations served as the foundation for comprehensive evaluation and interpretation.

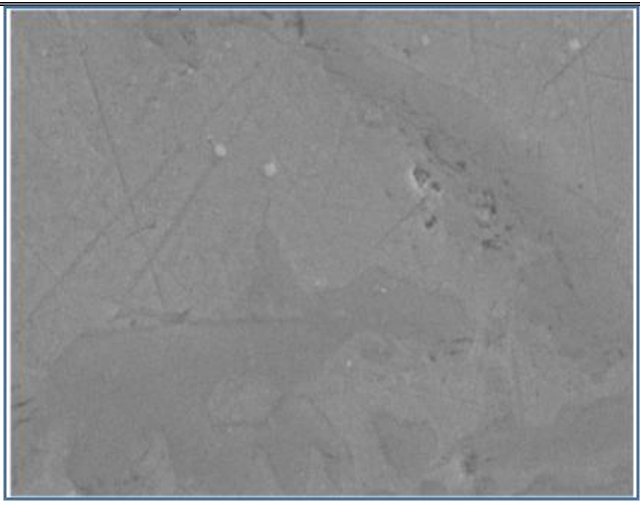

Figure 1. SEM micrographs of the synthesized composite specimen

The SEM images presented in Figure 1 clearly reveal the microstructural features of the fabricated composite material. These micrographs provide critical insights into the continuity of the alloy matrix and the spatial distribution of the reinforcement phases [15]. Through detailed observation, it was confirmed that the silicon reinforcement particles were uniformly dispersed within the matrix phase. Such a homogeneous distribution is primarily attributed to the effective preparation of the powder blend and the careful optimization of the sintering parameters. Correspondingly, previous studies reported in the literature emphasize that the even distribution of reinforcing phases within the microstructure is strongly dependent on the mixing uniformity during powder preparation and precise control over sintering conditions [15].

In addition to revealing the desirable microstructural traits, the SEM analyses also identified certain microstructural defects present in the samples. Notably, localized crack formations and porosity were observed within the microstructure. These defects typically arise due to incomplete densification of powder particles or suboptimal control of sintering temperature and atmosphere. Interestingly, a discernible reduction in porosity was correlated with increasing silicon content, suggesting that the presence of silicon influences the microstructural evolution during sintering. This phenomenon can be explained by the melting behavior of silicon particles during sintering and their interaction with the matrix, leading to microstructural rearrangements that effectively reduce pore volume [16 and 17].

Moreover, the SEM observations provided valuable information regarding the morphology of the silicon particles used as reinforcement. The silicon phase predominantly exhibited irregular, angular geometries with relatively consistent particle size distribution. This morphological characteristic is attributed to the mechanical milling process employed for particle preparation. It is well-established that particle morphology significantly impacts microstructural integrity, sintering kinetics, and ultimately the mechanical properties of the composite. The presence of sharp-edged particles may hinder adequate bonding with the matrix during sintering and can serve as stress concentration sites, potentially initiating localized mechanical failures. Therefore, controlling the morphology of reinforcement particles is crucial for enhancing the mechanical performance and reliability of the produced composites [18 and 19].

5. CONCLUSION AND RECOMMENDATIONS

In this study, silicon (Si) was incorporated as a reinforcing element into a high-entropy alloy (HEA) matrix to fabricate composite samples via powder metallurgy techniques. Throughout the manufacturing process, experimental parameters were maintained constant; compaction was conducted under a uniaxial pressing pressure of 600 MPa, followed by a sintering treatment performed at 1000 °C for three hours within a controlled atmosphere furnace. These processing conditions were selected based on precedent studies reported in the literature, aiming to ensure sufficient diffusion between the matrix and reinforcing phases, as well as to maximize densification of the material. Microstructural characterization of the fabricated composites was carried out using scanning electron microscopy (SEM). Evaluation of the obtained micrographs revealed that the overall structural integrity of the microstructure was preserved following the sintering process; however, localized regions exhibited crack formation and porosity. Such microstructural defects are commonly attributed to incomplete densification during sintering and inadequate bonding at particle interfaces. Comparative analyses further demonstrated that the porosity level varied as a function of the Si reinforcement content. An increase in Si concentration corresponded to a reduction in porosity, which is hypothesized to result from the near-melting behavior of Si particles during sintering, facilitating pore filling and microstructural rearrangement. Moreover, the Si particles were observed to be homogeneously dispersed within the matrix phase. This uniform distribution reflects the efficiency of the powder mixing process and the suitability of the sintering parameters, contributing positively to the isotropic mechanical properties of the final composite material. These findings align well with previously published studies on similar composite systems, reinforcing the notion that the homogeneity of the reinforcing phase within the microstructure is strongly dependent on meticulous control of powder preparation and sintering conditions.

NOTICE

This article is based on PhD thesis, entitled "Production and Characterization of Reinforced CrMnFeCoNi High Entropy Alloy", a thesis which was carried out under the supervision of Assoc.Prof.Dr. Mehmet AKKAŞ at Kastamonu University, Kastamonu, Türkiye.

CONFLICT OF INTEREST

The author(s) declare that they have no potential conflict of interest.

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DECLARATION OF ETHICAL STANDARDS

The authors of the article declare that the materials and methods used did not require ethics committee approval and/or regulatory approval.

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