

# Enhancing the properties of HEAs-ceramic composites using SPS technique

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**Abstract.** This study aims to evaluate the properties of High Entropy Alloy (HEA)-ceramic composites produced with spark plasma sintering (SPS) technique. HEAs are alloys consisting of five or more principal elements. They possess excellent mechanical, thermal, wear and corrosion properties as a result of their configurational characteristic effects. However, these properties usually attenuate at elevated temperatures. So, incorporating ceramic reinforcements enhances those unique properties at elevated thermal and mechanical stresses. SPS, a non-conventional sintering technique, offers unique properties to composite materials more than the conventional techniques. It prevents grain growth and evolution of deleterious intermetallic compounds and promotes rapid densification with strong interfacial bonding. The study systematically reviews HEA-ceramic composites developed with SPS, their properties, strengthening mechanisms, and property tailoring strategies. It also analyses how SPS parameters impact on the properties of the composites. Results show that SPS is a sustainable consolidation method, which can produce HEA-ceramic composites with superior properties suitable for demanding applications in energy, aerospace and biomedical sectors. The review highlights the novelty of incorporating ceramics into HEAs using SPS technique. It concludes by recommending optimization of material design and development strategies for next-generation robust materials.

## 1 Introduction

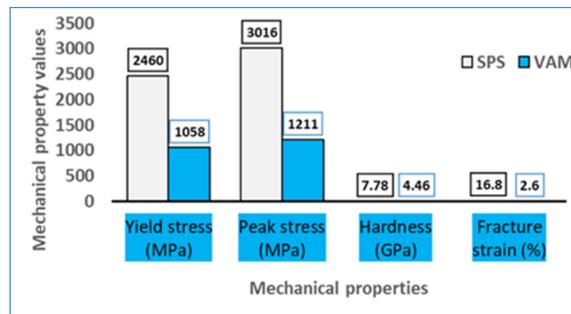
High entropy alloys (HEAs) are class of advanced materials that consists of multiple principal elements (5 or more) combined in equi-atomic composition; and was developed by Cantor and Yei in their independent research in 2004 [1]. HEAs include Cantor (CoCrFeMnNi) and non-Cantor based HEAs (CoCrFeMnNiAl, CoCrFeMnNiMg, etc), which are applied in automobiles, aerospace, defense, marine and energy sectors. They are characterized by superior mechanical, thermal and wear properties. HEAs find applications in aerospace, marine, energy, defense and automobile sectors. Conventional alloys, unlike HEAs, are made up of 1 or 2 principal elements. Even though HEAs have good properties as mentioned, they fail in some critical and harsh conditions. Hence, their optimal functionality needs to be augmented in order to expand their level of resilience and reliability. One of the most

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effective approaches for enhancing HEAs is by the incorporation of ceramic phases to create HEA-ceramic composites which can achieve unique hardness, good thermal stability and high wear resistance [2]. The main difficulty in developing HEA-ceramic composites consists of both achieving high densification and preserving their microstructural integrity. The traditional sintering techniques lead to deterioration of material performance as a result of grain coarsening and non-homogenous dispersion of reinforcements which leads to diminished mechanical strength as shown in Figure 1 [3]. The properties obtained from traditional sintering promote deterioration of mechanical, thermal, electrical and tribological properties of alloys and composites. However, with spark plasma sintering (SPS), which is the non-conventional sintering technique, these defects are avoided as it improves grain refinement, strong interfacial bonding and dispersion of reinforcement. In Figure 1, there is a comparison between spark plasma sintering and vacuum arc melting. As shown, the mechanical properties of spark plasma sintered samples were better than those prepared with vacuum arc melting.

Spark plasma sintering (SPS) is a non-conventional powder processing technique that utilizes pulsed direct current to densify power particles. It is rapid in operation with high heating rate and short holding time. It can produce composites within couple of minutes with high densification, minimal to zero grain growth and enhanced dispersion of reinforcements in the composite [4]. This research evaluates SPS effects on HEA-ceramic composites, focusing on their microstructure, mechanical, thermal and wear enhancement.



**Fig.1.** Comparing the mechanical property improvement of spark plasma sintering (SPS) and vacuum arc melting (VAM) of HEAs [3].

However, microstructural modification is an essential method of improving the functionality and reliability of HEA-ceramic composites. Through manipulating the grain particles, phase dispersion, micropores, and grain boundaries, the composite properties can be optimized to suit required usage [5]. Microstructures with refined grains exhibit high strength and hardness because of their large grain boundaries density. Also, microstructure with uniformly dispersed ceramics has high resistance to local stress concentration and crack initiation/propagation. Finely controlled interfacial bonding between HEA matrices and ceramic reinforcement possesses an adequate load transfer mechanism which enhances the fracture toughness and thermal properties of the resulting composite [6]. Moreover, optimally tailored microstructure possesses effective diffusion channels for particles with high oxidation resistance at elevated temperature. These are some of the significances of controlling the microstructure which can be made possible using advanced sintering technique of SPS. Hence, the use of the non-conventional sintering techniques (SPS) perfects accurate and desired microstructural tailoring through minimization of grain growth and retention of most desired phases [4]. Therefore, tailoring the microstructure functions as the principal linkage between composite design and its functionality, advancing the creation of the future, sustainable and robust HEA-ceramic composites.

## 1.1 Study rationale and objectives

HEAs are exceptionally unique material with excellent mechanical strength and thermal stability because of the constituent multiple elements, for example, FeNiCuTiMo, CuMgNiTiMo,  $(\text{TiTaNbNiMo})_x\text{Al}_{1-x}$ , FeNaNbTaTiNi, etc. Nevertheless, at some applications, their properties may experience some limitations, such as loss of strength, high temperature oxidation and grain growth at extreme temperature [7]. So, the addition of ceramic phase to the HEA matrix has been projected as an innovative technique to suppress the above limitations and enhance high temperature stability, oxidation resistance, fracture toughness and durability [2]. So, consolidating HEAs and ceramics with SPS has been presented to be a sustainable material for use in aerospace, biomedical, energy, defense and automotive industries. Hence, this study's rationale stems on the need to explore the role of ceramics and SPS technique in controlling the microstructure of HEAs and enhancing their properties, enabling their use in various demanding applications.

The novelty of the study, however, dwells on the systematic combination of the impacts of SPS technique and ceramic materials in enhancing the properties of HEAs. It was observed that previous studies have dwelt on either SPS of HEAs, or ceramics composites processed with SPS. Study that combines both materials and the processing technique is limited in the literature. So, this study systematically investigates the effect of ceramic type, distribution, grain boundary characteristics, and SPS processing parameters on the properties of the HEA-ceramic composites, thereby presenting a novel knowledge in the advancement of sustainable material for the harsh environment and critical demands.

The principal aim of this study is to systematically investigate the present trends in the advancement of sustainable new material (HEA-ceramic composites) produced through SPS processing method. This aim will be achieved via analyzing how various ceramic phases, SPS parameters, and interfacial bonding mechanisms affect the structural and functional properties of the composite. The study will equally showcase the comparative advantages of SPS over other conventional sintering techniques. Therefore, this research is poised to review the available relevant studies in literature, discover gaps and create new research outlook by establishing an optimal strategy for developing HEA-ceramic composites for sustainable and smart applications.

## 2 Role of ceramics on HEA improvement

HEAs have experienced major performance improvements when ceramic phases like SiC,  $\text{Al}_2\text{O}_3$ , TiC and  $\text{ZrB}_2$  are added to the matrix. The desired characteristics of improved hardness, strength, wear resistance and thermal stability emerge from the combination of HEAs and ceramics [2].

One of the main functions of ceramic phases in HEAs is to enhance the matrices' strength through the stress transfer kinetics and grain boundary pinning mechanisms [8]. The combination of ceramics like SiC and TiC with HEAs gives them exceptional hardness and elevated Young's modulus [9]. Ceramic particles embedded in HEA matrix operate as the stiffer components that absorb most of the mechanical stress unto their more resistant surface. The presence of ceramic particles, being the load-bearing component of the composite, improves both strength and hardness through effective load distribution which promotes resistance to plastic deformation of the system [10]. Another crucial impact of ceramic phase in HEAs is that of refinement of the grains. During processing using effective consolidation process like SPS, ceramic particles help in pinning the grain boundaries, thereby preventing grain coarsening [11]. From Hall-Petch relationship, it was established that refined grains contribute to increased mechanical strength. Also, particles of TiC and  $\text{ZrB}_2$  are especially

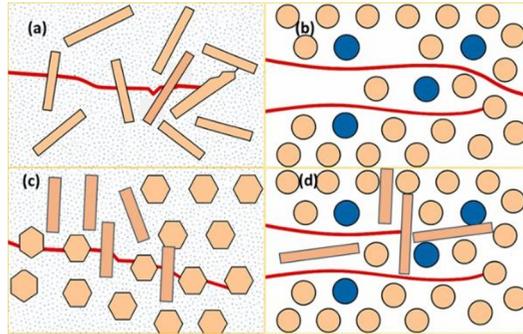
efficient in providing refined microstructures of HEAs-ceramic composites, translating to excellent mechanical strength with considerable ductility [12].

Furthermore, in HEAs composite systems, it has been observed that ceramic phases improve both wear and oxidation resistance. Ceramic phases like SiC and Al<sub>2</sub>O<sub>3</sub> possess good friction and wear characteristics, providing HEA-ceramic composites with excellent friction and wear resistance, and resistance to high temperature oxidation [13]. It was reported that ceramic particles usually form shielding oxide films to prevent high temperature oxidation, thereby improving the composites' resistance to elevated temperature corrosion. This high temperature resistance to oxidation is specifically important for the composites' application in harsh media like energy and aerospace sectors. HEAs reinforced with ceramics particulates have been proposed to be thermally stable at elevated temperatures. This is because ceramics have high melting temperatures, and their coefficient of thermal expansion (CTE) is very low. So, incorporating them into HEA matrix would improve the composites' mechanical and thermal stability, inhibiting high temperature softening or deformation by creep. It was reported that ZrB<sub>2</sub>, which has very high melting point (3040°C) with good thermal conductivity, strikes balanced properties of good mechanical strength and thermal stability when used as a reinforcement for HEAs [14]. In all, the systematic incorporation of ceramic phases into HEAs conditions the composites' microstructural characteristics and the entire system properties, resulting in sustainable advanced materials with a combination of the characteristics of metals (toughness and ductility), and that of ceramics (hardness and thermal stability).

## 2.1. Properties of HEA-ceramic composites

The development of HEA-ceramic composites gained substantial attention because the material integrates excellent characteristics from each constituent composition. The properties of these materials include high strengths, good ductility and thermal stability accruing from HEAs and extreme hardness, wear and thermal tolerance from ceramic additive. The systematic combination of these two material classes leads to composite that exhibits superior mechanical properties than the properties of their individual components.

A significant property possessed by the composite is improved hardness and resistance to friction and wear [4]. Incorporating hard particles of ceramic materials into strong and ductile phase of HEA matrix results in production of a composite with unique hardness and excellent resistance to wear and friction. These attributes are orchestrated by the load-carrying capacity of ceramics [15], and their tendencies to inhibit plastic deformation in the matrix. By inhibiting dislocation trajectory, ceramic particles help in enhancing composite's hardness and wear resistance. HEA-ceramic composites, furthermore, have excellent fracture toughness [16]. It is on record that ceramics are intrinsically brittle, but when dispersed into ductile HEA matrix, the synergy results in a material with high toughness. As shown in Figure 2, their toughening mechanism comes in the form of crack bridging, deflection and particle pull-out. Excellent toughness of the composite is maintained when there is a strong bonding between the ceramic phases and HEA matrix with substantial wettability.



**Fig. 2.** Schematics of prevention and bridging of cracks by ceramic phases on HEA matrices, a) crack bridging by ceramic fibers, b) crack stoppage by ceramic particles, c) crack bridging and stoppage by ceramic fibers, d) crack deviation and stoppage.

Another important property of the HEA-ceramic composites is high strength at elevated temperature [17]. Most engineering materials suffer loss of strength from creep or thermal stress at the temperature ranges of the following: steel (300 - 400 °C), Al (150 - 200 °C), Ti (400 - 500 °C), Ni (800 - 1000 °C), polymer (50 - 250 °C). But due to the thermal stability of HEA and ceramic materials, the composite maintains structural and mechanical integrity at a high mechanical or thermal stress levels. This property is made possible by the sluggish diffusion inherent in HEAs [18] and high melting temperature of ceramics [2]. The high resistance to creep or thermal degradation is what makes HEA-ceramic composites the ideal material for application in harsh environments like nuclear reactors, aerospace and cutting tools. Moreover, HEA-ceramic composites have sound compressive strength and stiffness. The addition of ceramic phases into HEA improves the composite's elastic modulus and compressive stress, thereby creating a material with high rigidity and excellent strength [19]. This property makes the composite suitable for applications in structural engineering where load-carrying capacity and dimensional precision are of utmost importance. So, HEA-ceramic composites possess excellent framework for manipulating mechanical characteristics via optimizing the processing parameters, constituent elements and their volume fractions. Their improved mechanical properties (hardness, toughness, elevated-temperature strength, friction and wear resistance) have opened new vistas for sustainable industrial and engineering applications.

### 3 Impact of SPS on the improvement of HEA-ceramic composites

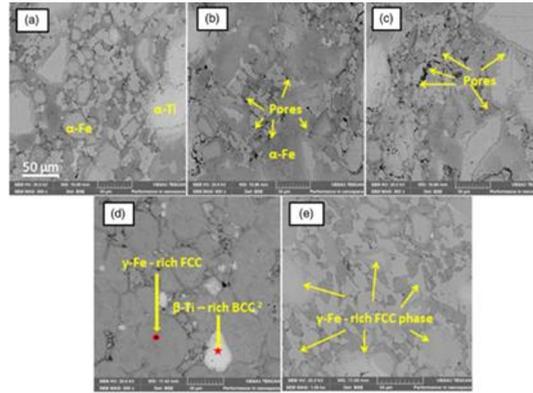
SPS technology stands as an efficient innovative powder consolidation method since its invention, and it surpasses other conventional sintering methods like pressureless sintering, hot pressing and hot isostatic pressing. Due to its advantages, SPS stands out in transforming high entropy alloy-ceramic composites and nanostructured powder materials into sustainable products.

Rapid heating rate is one of the most significant advantages of SPS over conventional sintering (CS) [4]. The rapid heating rate can be over 1000°C/min, and this is made possible via pulsed direct current which traverses through the powder inside the graphite die. However, the conventional sintering utilizes external heating components which creates slower heating rates that result in extended time of processing [20]. SPS's rapid heating rate helps to sustain fine nanostructures and metastable phases because it obstructs the growth of grains that occur in prolonged heating [18]. More so, SPS does its consolidation at a reduced sintering temperature and cycle time [3]. This is one of its importance over conventional sintering technique. With the aid of resistive heating on the powder particles, the required heat needed for effective densification of the powders tends to be lower than that required in

CS, which has no direct contact with the powder particles. The lower sintering temperature saves energy, sustains the environment and preserves the material from thermal degradation [21].

Moreover, the concurrent application of uniaxial pressure and pulsed DC enhances rapid densification efficiency [4]. While the pulsed current promotes joule heating and effective diffusion of particles, applied pressure boosts effective body contacts of the powder particles, neck formation and strong bonding [3]. But CS has the tendency of producing products with multiple pores, unless sintering is done at a very high temperature regime or long-time regime[20]. Furthermore, SPS has better control over the product's microstructure than the CS. This is because SPS does not permit adverse interfacial reactions nor grain coarsening [4]. It is for this property that the production of layered structures and functionally graded materials is readily done with SPS than with CS. The environment in which SPS is conducted (inert or vacuum) offers another advantage over that of CS. The inert environment prevents product degradation by oxidation. That is why it is used in production of reactive composites that are prone to high temperature oxidation. In all, the integration of high heating rates, uniaxial pressure, low sintering temperature, short holding time and inert environments makes SPS a better fabrication technique in powder metallurgy than CS for developing denser, purified, finer-grained and robust materials for advanced applications.

SPS improves the microstructure of HEA-ceramic composites via its inherent fast densification, grain refinement, and homogenous dispersion of reinforcement capabilities. These attributes are made possible by its high heating rate, short holding time and simultaneously applied heating and axial pressure which obstruct grain coarsening, eliminate micro pores, and improve strength and thermal properties [3]. The produced electric plasma coupled with localized joule heating in SPS enhance stronger inter/intra atom bonding between HEA and ceramics. Notably, improved microstructure translates to improvement in other properties of the composite making SPS a unique technique for processing high-performing materials with excellent properties for harsh environmental applications [22]. Meanwhile, optimization of the sintering parameters plays a vital role in microstructural improvement, phase evolution, mechanical and other property improvements as can be seen in Figure 3. Here, evolution of the dual FCC and BCC phases occurred at the optimal sintering temperature of 1000°C, exhibiting well refined microstructure [21]. This advanced processing technique has the capacity of improving the thermal stability of composites by ensuring that the porosity is eliminated in the microstructure. This is because pores are defects that can increase the electron, phonon and magnon scattering [23]. The presence of these pores reduces the thermal/electrical conductivity of the composites and by eliminating the pores conductivity can be enhanced. Moreover, SPS enhances the production of nano-scale precipitates and ceramic phases in HEA-ceramic composite, thereby increasing its oxidation resistance and thermal shock absorption [2].



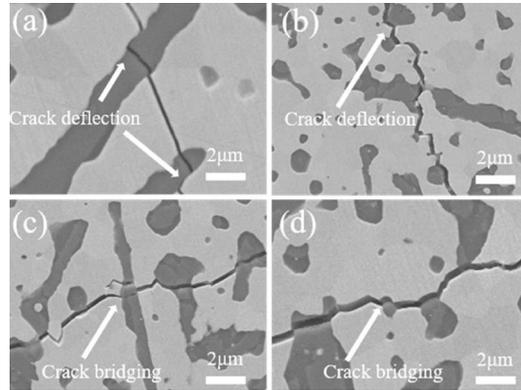
**Fig. 3.** Effect of sintering temperature on the phase evolution of Ti36-Al16-V16-Fe16-Cr16 HEA [21].

### 3.1 Toughening mechanisms of HEA-ceramic composites

In HEA-ceramic composites, the level of interfacial compatibility controls the mechanical properties as well as thermal/chemical stability characteristics of the material. In a structural material, there should be a strong bonding with high chemical stability between the matrix and reinforcements to obtain effective load transfer and avoid premature failure. When the matrix-reinforcement interfacial bond is weak, it generates concentrated stresses and facilitates initial crack formation that leads to delamination failures during mechanical or thermal stress [24]. So, manipulating matrix-reinforcement bonding reactions, differences in their coefficient of thermal expansion or thermal mismatch, and their homogenous solubility during processing, is very imperative for producing sustainable composites for advanced applications. These controls can be achieved with the use of advanced sintering methods like SPS [4]. The strengthening and toughening mechanisms witnessed in HEA-ceramic composites depend more on interfacial bond strength between these materials because it determines their ability to overcome brittle failure in ceramics [25]. Ceramic particles within the metal matrix cause approaching cracks to deviate their propagation path leading to energy dissipation that slows down the overall crack advances as shown in Figure 4. The mechanical properties of hard ceramic particles, being higher than that where cracks are propagating, would force the cracks to slow down, change their direction and lower their potential energy.

Crack bridging is another toughening mechanism in HEA-ceramic composites, as discussed in section 2.1. The toughening process via crack bridging becomes possible when ceramic particles dispersed on the HEA matrix bridge cracks to prevent their further opening (Figure 4) [2, 26]. The toughening effect is most successful when the ceramic phase bonds strongly with the HEA matrix, maintaining high interfacial bonding, even under mechanical or thermal stress. The selective failure of ceramic particles can occur through pull-out mechanism but only if the composite shows weak bonding at interfaces, and the outcome of loading would involve complete bond failure. Under certain conditions ceramic phases such as  $ZrO_2$  can transform because of stresses leading to toughening of the material. Here, localized volumetric expansion in the affected area squeezes crack at their tips and hinders their growth progression [27]. Also, thermal mismatch toughening can sometimes occur because HEAs have different thermal expansion with ceramic particles producing beneficial residual stress during the cooling process when stress control is optimized. In all, the degree of interaction between the toughening dynamics and the interfacial reactions in HEA-ceramic composites dictates the level of improvement in the composite system. So, the composite performance is improved by improving the interfacial compatibility through optimizing the process parameters, material selection, wettability and dispersion of ceramic phases in the

matrix. Hence, the need for advanced production techniques that will see to these property optimizations.



**Fig. 4.** Toughening mechanisms of HEA-ceramic composite, a-b) crack deflection, c-d) crack bridging [26].

### 3.2. Prospects and challenges of HEA-ceramic composites development with SPS

High entropy alloys integrated with ceramics unlock revolutionary opportunities in materials science that enable composites with outstanding mechanical properties alongside thermal durability and strong resistance to oxidation and material wear [3]. Through SPS technology, these composites achieve unprecedented consolidation outcomes. The rapid heating of SPS, its high densification efficiency, and limited grain growth allows the retention of essential nanostructured features necessary for remarkable composite performance [16]. HEA-ceramic composites produced by SPS methods possess characteristics that enable their use in aerospace, defense, biomedical and energy applications as they are capable of surviving harsh environments.

Meanwhile, a number of obstacles persist despite these potential prospects. The development of robust interfacial bonds between HEA matrices and ceramic reinforcements represents a complex technological challenge because thermal expansion mismatches and insufficient wettability can result in material failures [20]. The control of ceramic phase distribution to prevent their agglomeration remains challenging during production steps. Complex design processes and application-based optimization protocols are necessary during the selection of suitable ceramic materials and volume fractions to achieve specific properties. There is equally the challenge of accurate optimization of the SPS parameters as they play pivotal roles in controlling the microstructure and properties of the sintered composites [18]. An error in this stage could lead to disastrous product. SPS technology is still limited by scalability. This is because the initial cost of the equipment is still on the high side. However, the prospects of this process and material surpass the challenges. So, it is proposed that HEA-ceramic composites will advance to their next generation through computational modeling and precise process optimization for appropriate microstructural control of materials for sustainable and advanced engineering applications.

## 4. Conclusion and recommendations

The production of HEA-ceramic composites using SPS technique has made considerable progress in powder metallurgy and nanotechnology, creating a material with excellent integration of strength and ductility, hardness and toughness, wear and oxidation resistance, and thermal stability. This study discovered that even though HEAs have exceptional

properties due to their inherent configurational effects, those properties can even be more optimized and sustainable by incorporating ceramic phases into the matrix. The addition of ceramic reinforcements enhances hardness, toughness, chemical stability, oxidation stability, and wholesome structural integrity in the composite, making it the suitable material for harsh and demanding applications.

The impact of SPS technique on the HEA-ceramic composites are manifold. It enhances swift powder densification, suppresses grain growth, promotes refined microstructure, and promotes stiff interfacial bonding of the HEA matrix and ceramic reinforcement, creating composites very difficult to be developed with the traditional sintering. With the aid of the reinforcing phases and SPS technique, though with optimized sintering parameters, HEA-ceramic composites are toughened and strengthened through the grain refinement, crack deviation and crack bridging mechanisms. The new sustainable material from HEA and ceramics are prospectively useful in industrial and engineering applications such as aerospace, energy, biomedical and electronic components and devices.

It is then recommended that:

SPS operations shall always be preceded by optimizing the processing parameters as the best results can only be achieved with optimized parameters. More so, selection of ceramic types and their volume fractions must be guided by the specific application of the composite to be developed. For instance, incorporation of ZrB<sub>2</sub> should be for thermally demanding devices, TiC should be for components that need toughness, BN should be for applications where friction and wear are of great concern as it lubricates the interface. Optimizing the volume fraction of the ceramic is very crucial as this is influential to its wettability, homogenous dispersion, and interfacial bonding between the matrix and the reinforcement. Pilot testing in high temperature turbines or energy storage devices is recommended to test the composite materials' reliability, resilience and sustainability in the real-world.

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