A study of silicon carbide reinforced W-Ni-Cu based heavy alloys sintered with different heating modes

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Abstract

The influence of silicon carbide (SiC) addition to W-Ni-Cu-based heavy alloys has been investigated in the present study. The powders of W-Ni-Cu with varying percentages of SiC were blended and sintered using conventional and Spark Plasma Sintering (SPS) techniques. The sintered samples were characterized to determine the density, microstructure, and mechanical properties. The alloy (W-7Ni-3Cu-0.5SiC) exhibits high ultimate tensile strength 430 MPa for conventional sintering and 831 MPa for spark plasma sintering, relative sintered density 71.84% and 88.25% of conventional sintered and spark plasma sintering, respectively. After the tensile test, the fracture surfaces show a mixed-mode fracture consisting of brittle W/W intergranular and ductile mode of fracture in the matrix.

1. Introduction

Tungsten heavy alloys (WHA) exhibit a unique combination of high density, strength, and toughness [1], and hence find complete applications in defense [2] and aviation industries [3]. The unique combination of BCC phase (W) and FCC (Ni and Cu) matrix provides high mechanical properties such as strength and elastic modulus and high absorption capacity of X-rays and γ-rays [2, 4]. W/W contiguity, W/matrix interface interaction, and matrix volume fraction are the critical factors for improving mechanical properties [5]. Processing W has been a challenge from the very beginning [6] due to its very high melting temperature (3200 °C), making it very difficult to sinter with high density. Therefore, WHAs and advanced processing techniques are used to process them at reasonable lower temperatures. The Bulk Self diffusion coefficient of tungsten is very least, so it is difficult to sinter alone. W and Cu are soluble in Ni at high temperatures, which is favorable to the formation of Ni–W and Ni–Cu solid solution. Ni has a potential additive to improve the wetting and adhesion of Cu and W, further enhancing the densification [7]. The J Kurtz et al (1946) [5] work addition of the Ni to tungsten improves the grain boundary self-diffusion coefficient, and large tungsten single crystals turned into the matrix of the fined grained structure. X Xu et al (1999) [8] stated that the addition of the copper to the tungsten showing the low mutual solubility and tungsten particles were not dissolving in the liquid copper, resulted in the low sintered density(70%) but from the Wang et al (2002) [9] study addition of the two wt% copper to the W-7Ni-3Fe alloy showing the improvement in the sintered density(90% to 99%) and bending strength(359 MPa to 576 MPa). From the above two studies, we observed that only W with Cu results in fewer mechanical properties and sintered density compare with the W-Ni-Fe-Cu alloy. W-Ni-Cu alloys are generally sintered via liquid phase sintering (LPS), as Ni-Cu turns into a liquid phase during sintering and acts as a binder and facilitator to assist sintering. The main advantage of Ni-Cu based tungsten heavy alloys is that they...
can be sintered to almost 90% of theoretical density in the temperature range of 1100° – 1300 °C [10–13]. During the last few decades, Spark Plasma Sintering (SPS) technique has been used to consolidate various metals and ceramics due to its rapid heating rates and high densification of the material even at the lower temperatures [14–16]. SPS also offers considerably reduced sintering time and fine microstructure. SiC is one of the non-oxide ceramic. It has many attractive properties like a high melting point, specific strength, corrosion, less density, and erosion resistance [17]. SiC and W were mainly used in the filaments and semiconductors previously due to the limited technology. Now SiC operates in the heat exchangers, cutting tools, automotive water pump seals, and sandblasting injectors [18]. SiC has an attractive feature suitable for structural applications. Significantly less work is done on the W-SiC composite, and W and SiC have almost the same coefficient of thermal expansion [19]. Kang et al interpreted a small SiC special additive amount to toughen the tungsten [20]. William et al stated that carbide and silicide were both the most stable configurations are formed with W; silicide is formed WSi2 and W2Si3, carbide will include WC and W2C, based on the different temperature carbides are comprising [21] another author Chengcheng et al illustrated that W reacts with the SiC and form WC, W2C and WSi2 [22]. Hot pressed W-SiC composite has positive bond strength in between the W and SiC [23]. This carbide may influence the mechanical properties. W-4Wt%SiC has 97.7% relative sintered density, and 93 GPa micro Vickers’ hardness is achieved [24]. In the present study, an attempt has been made to sinter WHA + SiC at a temperature of 1050 °C using conventional vacuum sintering and SPS techniques. A comparative analysis of the results of the two approaches has also been made.

2. Experimental procedure

As-received W, Ni, Cu, and SiC powders (Supplier: Sigma Aldrich, Purity:99.5%) were blended in the desired stoichiometry to compare particulate reinforcement’s effect the different heating/sintering modes. Figure 1 shows the morphology in the SEM micrographs of all as-received powders. The average particle size of the W and SiC powder ranges between 10–12 μm, whereas for Ni and Cu, it ranges between 2–3 μm and 100–120 μm, respectively. The powders were mixed in 90W-7Ni-3Cu proportion, with a varying percentage, i.e., 0.5, 1, 1.5, and 2 wt% of SiC. To ensure homogeneous mixing of the powders, dry powders are blended in a planetary ball mill (without balls and no medium) for an hour. The mixed powders for conventional sintering were cold
compacted in 30 mm (internal) cylindrical die using a uniaxial hydraulic press with a pressure of 600 MPa and sintered in a vacuum ($10^{-5}$ mbar) furnace at 1050 °C with a heating rate of 5 °C min$^{-1}$ and 1 h dwell time. To do the Spark Plasma Sintering 30 mm internal diameter cylindrical graphite dies used in this study. Before pouring the powder into the die, the graphite foil is placed in the die to remove the sample after sintering easily. After pouring the required amount of powder into the die, place the die in the SPS furnace [25]. For the SPS method, Dr. Sinter 21050 furnace was used and sintered at 1050°C with a heating rate of 100 °C min$^{-1}$ under 30 MPa pressure and 1 min dwell time. The temperature was determined using an IR pyrometer. The sintered samples are cylindrical with a 30 mm diameter. SPS and conventional sintered samples’ relative sintered densities are measured from the theoretical density and actual density. The theoretical density is calculated using the

Figure 2. SEM (BSE-Back Scattered Electrons) micrographs of (A)-Conventional sintered samples and (B)-Spark Plasma Sintered samples.
where \( \rho_i \) and \( w_i \) are theoretical density and weight fraction of the \( i \)th element, the Actual density is measured according to the Archimedes principle. The sintered samples were cleaned and prepared for microscopic examination by doing grinding and fine polishing using the aqua-alumina solution. All samples were etched using Murakami’s Reagent, as mentioned in ASTM E-407. To analyze the morphology of powders, Microstructure & EDS analysis, and fracture behavior of the broken tensile test specimen Scanning Electron Microscopy (FE-SEM, Quanta 250) were used in this study. To identify the interaction of alloying elements with base metal, high-resolution SEM imaging was done. In EDS analysis, 200 \( \mu \)A emission current and 20 kV accelerating voltage used, which means the energy of electrons in the primary beam is 20 keV. After the microscopic examination, the same sample’s micro-hardness is measured using Vickers’s micro-hardness tester (Leco-Microhardness tester), with the load is 0.5 kgf 10 s of dwell time. Dog bone shaped Micro tensile sample is used for tensile testing; a micro tensile sample is prepared according to the ASTM E-8 standard dimension. Tensile testing is carried out in the universal testing machine (Instron 8801) with a strain rate of 0.2 mm min\(^{-1}\). The fractured surfaces were also examined to study the nature of the fractures.

### Table 1. Percentage of sintered density achieved in both Conventional and SPS sintering samples.

| Composition          | Green density | Sintered density | SPS. sintered |
|----------------------|---------------|------------------|---------------|
| 89.5W-7Ni-3Cu-0.5SiC | 64.73 ± 0.5   | 71.84 ± 1        | 88.25 ± 2     |
| 89W-7Ni-3Cu-1SiC     | 60.58 ± 0.2   | 69.43 ± 0.5      | 82.57 ± 0.7   |
| 88.5W-7Ni-3Cu-1.5SiC | 58.78 ± 0.9   | 65.24 ± 0.6      | 78.05 ± 0.5   |
| 88W-7Ni-3Cu-2SiC     | 54.89 ± 1     | 56.84 ± 0.4      | 75.01 ± 1     |

### Table 2. Vickers microhardness of conventional and SPS sintered W-7Ni-3Cu with varying % of SiC.

| Composition          | Conventional sintered | SPS. sintered | Conventional sintered | SPS. sintered |
|----------------------|-----------------------|---------------|-----------------------|---------------|
| 89.5W-Ni-Cu-0.5SiC   | 0.61 ± 0.09           | 0.46 ± 0.11   | 138 ± 10              | 263 ± 15      |
| 89W-Ni-Cu-1SiC       | 0.72 ± 0.12           | 0.42 ± 0.14   | 148 ± 9               | 268 ± 10      |
| 88.5W-Ni-Cu-1.5SiC   | 0.66 ± 0.2            | 0.51 ± 0.1    | 155 ± 12              | 296 ± 11      |
| 88W-Ni-Cu-2SiC       | 0.65 ± 0.16           | 0.44 ± 0.08   | 165 ± 14              | 322 ± 14      

Below formula.

\[
\rho_{\text{theoretical}} = \sum (\rho_i \times w_i)
\]

Figure 3. SEM (BSE) micrographs of SPS sintered 90W-7Ni-3Cu-0.5SiC. 3 Phases identified. [A]-Tungsten grains (white), [B]- Ni-Cu Matrix (light grey), [C]- W-Ni (dark grey), [O]- oxide impurity formed during cooling.
3. Results and discussions

3.1. Effect on densification

The sintered densities of conventionally sintered and SPS sintered W-Ni-Cu-SiC composites are presented in Table 1. SPS sintered samples have achieved higher densities for all compositions when compared to conventionally sintered corresponding samples. A maximum sintered density of 88.25% in the SPS method was performed with 0.5% SiC while the conventionally sintered sample gave 71.84% density. Mondal [27] demonstrated that the sintered density of 90W-7Ni-3Cu in conventional and microwave sintered alloy was 69 ± 2% and 67 ± 2%, respectively. The sintering temperature of both methods used by Mondal was 1200 °C [27]. In SPS, high densification is attributed to the application pressure and temperature at a time. Furthermore, due to localized sparks at the grain boundaries, the neck formation and growth occur at a lower temperature than the conventional sintering process. The neck growth stage is assisted by high pressure through enhanced...

Table 3. Effect on ultimate tensile strength and % Elongation of conventional and SPS sintered W-7Ni-3Cu with varying percentage of SiC.

| Composition          | Grain Size (μm) | UTS (MPa) | % Elongation | Grain Size (μm) | UTS (MPa) | %Elongation |
|----------------------|-----------------|-----------|--------------|-----------------|-----------|-------------|
| 89.5W-7Ni-3Cu-0.5SiC | 12 ± 1.6        | 430 ± 5   | 1.37 ± 0.2   | 4.68 ± 0.41     | 851 ± 8   | 1.11 ± 0.12 |
| 89W-7Ni-3Cu-1SiC     | 14.47 ± 1.44    | 355 ± 7   | 1.4 ± 0.4    | 5.72 ± 0.23     | 707 ± 7   | 1.7 ± 0.23  |
| 88.5W-7Ni-3Cu-1.5SiC | 15.35 ± 1.52    | 256 ± 4   | 1.04 ± 0.32  | 6.27 ± 0.36     | 440 ± 9   | 1.36 ± 0.12 |
| 88W-7Ni-3Cu-2SiC     | 16.48 ± 2.06    | 232 ± 6   | 1.21 ± 0.2   | 8.87 ± 0.48     | 358 ± 11  | 0.95 ± 0.31 |

Figure 4. EDAX analysis of SPS sintered 89W-7Ni-3Cu-1.0 SiC. W–Tungsten, Ni–Nickel, Cu–Copper, and O–SiC.
mass transportation [28]. Grain boundary diffusion is also believed to play a predominant role in densification, as K. Hu [29]. K Hu has also stated that Ni tends to behave like an activator in W densification. W and Ni have good solubility. Ni and Cu also have mutual solubility in solid as well as in liquid states. However, W has no solubility in Cu. The Cu is susceptible to a melt since the sintering temperature is very close to Cu’s melting temperature (1085 °C). In such a case, Ni will readily dissolve in Cu melt, and both form a smooth flowing binder phase. These result in superior densification that is assisted by the pressure used in the SPS process. The overall densification mechanisms are attributed to dissolution and precipitation through the binder phase, W-W grain coalescence, and surface diffusion at the W-Ni-Cu matrix boundary [30]. Another factor attributed to densification and grain-growth is the phenomenon called electro-migration. The enhanced neck growth is due to electric currents and resulting in electro-migration [28]. It is also evident that the SiC addition has no role in assisting the sinterability. It is also observed that the sintered density decreases with an increasing amount of SiC addition due to its non-wettability with the W or binder phase.

3.2. Effect on microstructure

The SEM images of the conventionally and SPS sintered samples are displayed in figures 2 and 3, respectively. In both cases, three distinct phases are identified: white W-grains, light grey Ni-Cu matrix phase, dark grey W-Ni binder phase, and various SiC particle distribution throughout the microstructure. This three-phase microstructure is similar to the one observed by Ding et al [31]. The conventional and spark plasma sintering microstructures clearly showing the difference. Traditional sintered compact’s microstructure tungsten rich grains and binder grain size are high due to the increased dwell time. In the case of the SPS compacts, the microstructure’s small tungsten grain and binder phase are uniformly distributed. Due to the high dwell time in the conventional sintering tungsten grain size is increased, in SPS dwell time is very least, there are fewer chances to increase the grain size. EDAX analysis (figure 4) of samples reveals pure tungsten grains. The Ni-Cu matrix is observed clearly due to its mutual solubility, as expected. SiC particles are observed to spread continuously throughout the Ni-Cu matrix. However, in some areas, EDAX reveals that SiC has been converted into the SiO2 phase. The Ni and Cu have mutual solubility in solid as well as liquid phases. The W and Ni have solubility as well. In contrast, the W and Cu have no solubility at all. Conventionally sintered samples show coarse grain structure due to the long sintering cycle time in conventional sintering. The grain growth in SPS sintered samples is limited due to the short sintering cycle time. SiC addition does not play any role in controlling grain growth.

The contiguity and hardness values of conventionally and SPS sintered samples are listed in table 2. The SPS samples have contiguity in the range of 0.4–0.5, following Das [32], whereas the conventionally sintered samples give high contiguity between 0.6 to 0.7. The increased contiguity is associated with reduced ductility [33]. With increasing SiC addition, the overall % elongation values are decreased. This trend is observed for both cases; conventional as well as SPS processes. The volume fraction calculations were not determined due to the high inconsistency in the phase’s distribution. The sharp edges of the grains suggest that no liquid phase of tungsten has formed in figure 3. In the case of liquid phase formation, tungsten re-precipitation occurs, and tungsten

### Table 4. Comparison of Previous results with the present work.

| Composition       | Tensile Strength (MPa) | Grain Size (μm) | Relative Sintered Density (%) | Sintering Temperature (°C) | Process        |
|-------------------|------------------------|-----------------|-------------------------------|-----------------------------|----------------|
| 94.9W–3.4Ni–1.7Cu [34] | 655                    | 60              | —                             | 1500                        | LPS & Heat     |
| 92.5W–5Ni–2.5Cu [34]  | 681                    | 30              | —                             | 1500                        | LPS & Heat     |
| 96.1W–2.8Ni–1.1Cu [34] | 670                    | 70              | —                             | 1500                        | LPS & Heat     |
| 95W–3.5Ni–1.5Cu [35]   | 660                    | 60              | —                             | 1460                        | LPS            |
| W–72Ni–24Cu [36]       | —                      | 23.1 ± 1.3      | —                             | 1500                        | L.P.S.         |
| W–12Ni–8Cu [37]        | —                      | 16.5 ± 4.5      | —                             | 1400                        | LPS            |
| W–14Ni–6Cu [37]        | —                      | 24.5 ± 2.5      | —                             | 1440                        | LPS            |
| W–16Ni–4Cu [37]        | —                      | 33.5 ± 5.8      | —                             | 1480                        | LPS            |
| 90W–7Ni–3Cu [27]       | —                      | 23 ± 10         | 68 ± 1.9                      | 1450                        | Conventional   |
| 90W–7Ni–3Cu [27]       | —                      | 21 ± 7          | 71 ± 1.2                      | 1450                        | Microwave      |
| 89.5W–7Ni–3Cu–0.5SiC (Current Work) | 430 ± 5              | 12±1.6          | 71.84 ± 1                    | 1050                        | Conventional   |
| 89.5W–7Ni–3Cu–0.5SiC (Current Work) | 851 ± 8              | 4.68 ± 0.41     | 88.25 ± 0.2                  | 1050                        | S.P.S.         |
particles start dissolving in the binder phase and result in smooth edges of grains [29]. The absence of soft edges and no visible spheroidization suggest no liquid phase formation of tungsten.

The hardness data of conventional and SPS samples are displayed in table 2. In the case of conventionally sintered samples, the hardness data shows an increasing trend with the amount of SiC, from 138 Hv (0.5 wt% SiC) to 164.76 Hv (2 wt% SiC). In the case of SPS samples, an even higher increase in hardness is obtained, from 263 Hv (0.5 wt% SiC) to 322 Hv (2 wt% SiC). In both cases, the increase in hardness is attributed to grain size and contiguity. The grain sizes of the conventionally and SPS sintered samples are displayed in table 3.
In conventional sintering, the grains have enough time to grow, leading to low mechanical properties. However, in SPS, the short sintering cycle time and the short dwell time gave finer microstructure. The mean grain size of conventionally sintered samples is 14.57 μm, and in SPS, it is 6.38 μm. This reduction in grain size clearly explains the increase in corresponding values of hardness. The particulate reinforcement of SiC, which settles around W-grains, hinders the dislocation movement. It can be argued that since the density is less, there must be inherent porosity, which results in fewer particles to particle contact. This means that the alloy’s binding energy is low, and the indenter should penetrate easily. However, this claim can be countered by stating that SiC acts as particle reinforcement and hinders the dislocation movement.

The ultimate tensile strength (UTS) values of the conventional and SPS sintered samples are displayed in table 3. For SPS sintered samples, the maximum UTS value obtained is 851 MPa, whereas, for traditional samples, the maximum UTS value obtained is 430 MPa 0.5 wt% SiC compositions. Conventional sintered samples have low values due to the high sintering dwell on time, coarse grains, and less plastic flow. SPS samples have many more refined grains and hence exhibit much higher UTS values. Table 4 compares the current work properties with other W-Ni-Cu alloys fabricated in different processes at different temperatures. From table -4 data, it was observed that spark plasma sintered 89.5W-7Ni-3Cu-0.5SiC alloy has better stuff than the microwave sintered W-7Ni-3Cu [27] and other alloy shown in the table-4.

Figure 5 shows the SEM fractography of conventionally and SPS sintered samples, respectively. The mixed-mode fracture (ductile and brittle) is observed in both cases. The four primary methods of crack propagation are suspected: W-cleavage, W-matrix separation, W-grain boundary separation, and matrix rupture. Different types of fracture locations are identified in figure-5. In samples where density is less, the low particle-particle contacts prefer to undergo matrix rupture failure. This mode is observed more in the case of conventional sintered samples. SPS sintered illustrations show the predominantly mixed-mode fracture with W-grain boundary separation and intergranular fracture.

4. Conclusion

Conventional and Spark Plasma Sintering methods performed the sintering of 90W-7Ni-3Cu with incremental SiC addition. SiC was added in the amount of 0.5, 1.0, 1.5, and 2.0 wt%. A comparison of the results of conventionally and Spark plasma sintered alloys led to the following conclusions:

Compared to the conventionally sintered alloys, relative sintered density spark plasma sintered alloys has much higher relative sintered density. Silicon carbide addition above 0.5 wt% to W-Ni-Cu alloy does not show much effect in the densification.

Grain growth was significantly hindered due to reduced sintering cycle time and dwell time for the SPS processes. All the SPS sintered samples show a more refined microstructure than conventionally sintered samples. The minimum grain size is 4.68 μm obtained for spark plasma sintered 0.5SiC-90W-7Ni-3Cu compact.

The direct impact of grain size reduction is observed on hardness and UTS values. SiC addition also played an essential role in improving hardness by acting as particulate reinforcement. SiC particles within the Ni-Cu matrix hinder the dislocation movement, thereby improving hardness. The highest hardness value of 322 Hv is obtained for 88W-Ni-Cu-2SiC through SPS sintering.

It can be concluded that up to 0.5 weight percentage SiC addition improved the mechanical properties; compared to the conventional sintered and spark plasma sintered W-Ni-Cu/SiC composites, spark plasma sintered alloys have much better mechanical properties.

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