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Hot Explosive Consolidation for Nanostructured Tungsten-Silver Precursor Materials

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Abstract: The HEC technique was used to consolidate several precursors of refractory nanostructural tungsten-silver (W-Ag) composites into cylindrical billets. Consolidated to near theoretical density below and over the melting temperature of silver (940 - 1050°C), several compositions including a nanoscale W phase (100 nm) and a coarse matrix phase of Ag were tested. All of the tests had loading intensities of around 10GPa. High densities, excellent integrity, and outstanding electrical characteristics were achieved by consolidating nanostructural W-based composites using a combination of high temperatures and two-stage explosive densification methods. The value of the consolidation temperature and the diameter of the consolidated particle determine the structure and property of the samples. Samples without cracking, with a high density value, and uniform distribution of the two phases were produced for W-Ag based composites by applying high heat and consolidating precursors to the melting point of silver, which is 940 °C. Phase content and consolidation circumstances determine the aspects of the structure-property connection, which have already been covered.

Keywords: Coefficient of thermal extension, tungsten-silver composites, nanostructure, and hot explosive consolidation.

Introduction

Heavy alloys based on tungsten (W) have seen a rise in commercial and industrial use in the last few years. The majority of heavy alloys have W particles interspersed in a matrix of other metals or alloys, often iron, nickel, or copper.1 A few examples of possible applications for W-silver (Ag) composites include heat dissipation materials for microelectronic devices that fail at high operating temperatures, diverter plates for fusion reactors, and unique industrial uses (like aerospace) for this material.

Comparing the mechanical characteristics of samples made from nanocrystalline precursor powders with those of conventional, coarse-grained materials reveals striking differences, according to experimental investigations. A few examples of the remarkable features shown by these materials include their extreme hardness and wear resistance, their exceptional fluidity, and the perfect harmony between their strength and elasticity. The material's optical, thermal, electrical, and magnetic characteristics are enhanced when the average grain size is equal to or less than the wavelength of visible light. Improving the materials' characteristics might be as simple as reducing the grain size and controlling the fault substructure of the grains at the same time. Many different techniques exist for creating monolithic specimens from precursor powders with sizes ranging from micrometres to nanometers (i.e., covering the visible light spectrum from infrared to ultraviolet wavelengths) and other media.

Along with their beneficial effects, all conventional technologies now in use also bring about some undesirable side effects. Because grains on the nanometer scale are so heat sensitive, these powders expand at high temperatures. The unique physical and mechanical properties that would normally be inherent to nanostructural materials are lost in monolithic materials formed under these conditions because the grain growth is usually not uniform and its overall impact causes nanostructure imperfections and non-uniformity.

Reducing the sinter or compaction temperature during low-temperature processing usually does not provide the desired result. Here, high-density samples are unable to achieve due to the powder's relatively large free surface area. In addition, when refractory and ceramic powders are compressed and solidified, the necessary interfacial grain-to-grain barriers do not develop at low temperatures. Because of their high porosity, these samples lack sufficient physical or mechanical qualities.

Still, enough background knowledge exists to address some of the issues listed above. The plan is to subject the samples to very high temperatures (up to 1200 °C) in order to facilitate quick consolidation under these extreme circumstances. To make the sample more malleable, heat the powders or alloys

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before loading. This leads to the formation of interfacial solid solutions, shared borders, intermediate layers (when combining bulk alloys), and other useful properties. Grain growth processes are prevented or suppressed by the high value of the recrystallization temperature of W.

The two-stage process of consolidating solid samples in a cylindrical shape from sub micrometre- and nanometer-sized W-Ag mix powders is another unique aspect of the suggested unconventional method:

(a) In the first step, the precursor powder mix is subjected to a room-temperature explosive compression with a loading intensity of 5 to 10 GPa. This will raise the initial density of the blend and activate the surfaces of its particles.

(b) In the second step, the cylindrical sample, which has already undergone densification, is subjected to a main explosive shock wave again, this time with a loading intensity of 10GPa and a temperature ranging from 20 to 1100 °C.

The primary goal of the first consolidation step should be to compress the precursor powder without altering its microstructure, according to expectations. Using an electrical furnace, the pre-compacted samples undergo the second step of consolidation, which is hot consolidation. Preliminary experiments showed that W's high recrystallization temperature greatly reduces the likelihood of thermally activated grain growth, but high intensity shock wave compression makes the grains more plastic by adding fluidity to their surfaces; this, in turn, generates particle-particle bonds that wouldn't form under quasistatic conditions.

We used hot explosive consolidation (HEC) procedures to form cylindrical rods from combinations of tungsten and silver powder with a weight percentage of 10 to 50 percent. At various temperatures up to 1100 °C, two kinds of nanometer-scale W-Ag blend compositions were solidified to a density close to theoretical one, containing 100 nm W and 10 and 50% 5 μm Ag. Shockwave loading was around 10 GPa.

Research aimed to ascertain if satisfactory homogeneity, strong metal-particle bonding, and void-free products could be obtained by using high temperatures and two-stage shock wave processing. These microscopic characteristics would show how consistent the mechanical and thermal properties are. The mechanical parameters, including internal friction losses and elastic modulus, were also studied in relation to processing temperature, processing technique, and Ag content. Finally, electrical resistivity and diamagnetic susceptibility, two electromagnetic characteristics, were measured. Here we detail these outcomes.

Experimental

American Research Nanomaterials, Inc. (USA) supplied the W particles with a nominal nanoscale size, whereas Inframat "Advanced Materials" (Manchester, USA) supplied the coarse Ag powders employed in these research.

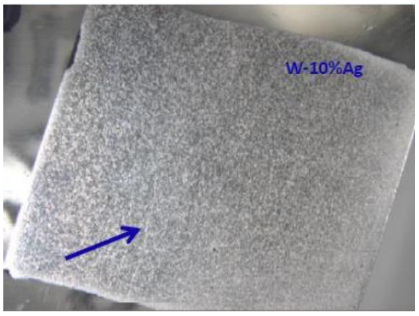
We used elemental powders that were already on the market to make the W-(10-50)%Ag precursor powder mixes. The next step was to use hot explosive consolidation (HEC) techniques to solidify the particles into cylindrical rods. Theoretical density was approached by consolidating three varieties of W-Ag. The diameters of the coarse Ag particles used in the studies are around 5 μm. Compaction temperatures varied, with a high of less than 1100 °C, and the shock wave loading intensity was around 10 GPa.

As mentioned before, the explosive compaction process is executed employing a cylindrical system of dynamic loading.4

Shock waves are usually set up by industrial explosives that have been mixed with different concentrations of ammonium nitrate.

Microhardness tests and microstructural analysis were performed on the HEC samples after consolidation. To find out whether the W-Ag composite might be used in an electronic assembly with other heat-dissipating materials like ceramics and semiconductors, the Coefficient of Thermal Extension (CTE) was determined. In addition to mechanical characteristics like elastic modulus and internal friction, electro-magnetic parameters like electrical resistivity and magnetic susceptibility were also studied.

Results and Discussion



The nanoscale powders indeed include particles on the nanometer scale, but they were highly agglomerated into multi-micrometer agglomerates, according to scanning electron microscopy (SEM).

Figure 1a and 1b show, respectively, a cross-sectional view of W-Ag billets made using two-stage HEC at 1050 °C with W-10%Ag nanometer-sized and W-50%Ag. The macrostructures of the samples were found to be significantly different upon comparison. While radial fractures are seen in both samples, an extra zone has developed throughout the billet's length in the middle of the one with a high silver content (1b). Overcompression and shock wave collisions in the sample's centre may explain the distinctive macrostructure (Figure 1b) of the newly-formed zone, which is color-different from the rest of the sample. The term "Mach's effect" describes this

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phenomenon.

a)

b)

Figure 1. The Optical micrographs view of HEC W–Ag samples consolidated at 1050 °C with 10GPa. a) W–10%Ag at 6×. Radial cracking is observed b) W–50%Ag at 6×. Compositional segregation and radial cracking are observed.

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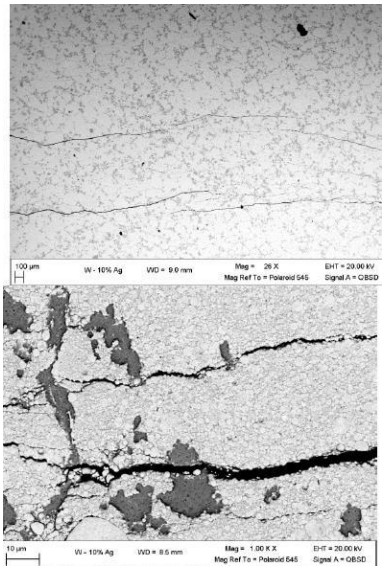
Figure 2 illustrates the microstructures of different parts of the W–10%Ag composition presented in the Figure 1. Extensive radial cracking was observed in samples of the W–10%Ag composite (Figures 2a). The explosive forming of the dense W–10%Ag composite achieved good bonding between W and Ag particles. This is shown in Figures 2b, taken at 100× and 1000× correspondently. As expected, the Ag particles, initially of 8 – 10μm in size, tended to agglomerate in nodules of approximately 30μm in size in average around large areas of W in the W–10%Ag sample.

a)



b)

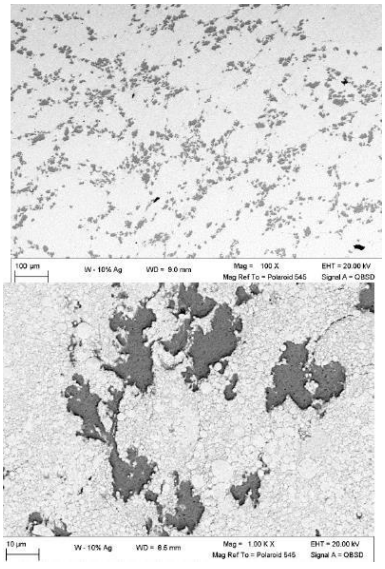


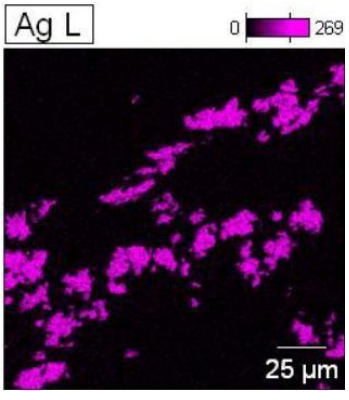
Figure 2. Microstructures of W–10%Ag HEC sample obtained at 1050 °C temperature with intensity of loading 10GPa. (a) Significant radial cracking is observed. (b) Distribution of Ag particles (in dark colour) – good bonding between W and Ag particles.

Hot explosive consolidation at 1050 °C, while the bonding phase is in a liquid condition, seems to have both positive and negative effects on high density W-10%Ag composites, leading to the creation of radial fissures. In reality, when the bonding phase is liquid, the liquid phase consolidates, allowing the Ag phase to be extruded onto the front of the shock wave during compression and improving the densification of

solid-state processes include the plastic movement of particle surfaces and the subsequent creation of joint boundaries, in contrast to solidified predecessors.

It is certain that solid solutions and novel chemical compounds may be formed under comparable loading circumstances thanks to the presence of a liquid bonding phase during consolidation under shock wave compression. However, high density billets may be readily manufactured and explosives with low detonation velocities can be used with longer pulse durations thanks to liquid phase shock wave consolidation. The compositional mapping of the HEC W-10%Ag sample, as shown in Figure 1a, is shown in Figure 3.

(a)



(b)

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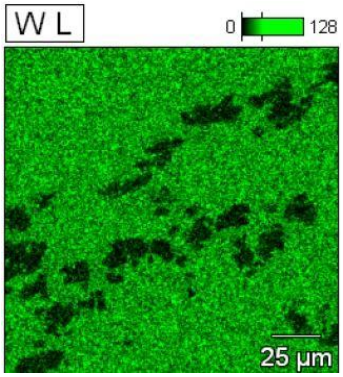

(a)

Figure 3. The location and distribution Ag (lavender color) and W (green color) in the matrix (grey colour) is shown.

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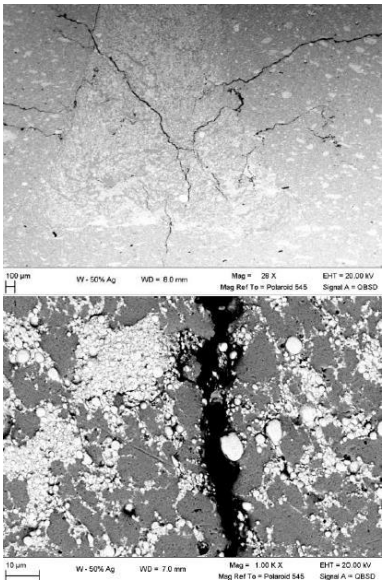
As mentioned above, distribution of phases of W–10%Ag showed that Ag precursor, initially of 8 – 10 μm in size, agglomerates into nodules of average size of approximately 15 to 30 μm.

The appearance of agglomerates of silver phase in some regions of HEC W–10%Ag may be explained as an influence of high temperature and low intensity of compression that may not provide good extrusion and uniform distribution matrix of silver phase in whole volume of obtained billets.

In order to evaluate influence of silver content in W–Ag phase on consolidation process, HEC method was applied under similar experimental conditions on W–50%Ag composites also. Figure 4 illustrates the microstructures of different parts of the W–50%Ag composition presented in the Figure 1b.

a)



b)

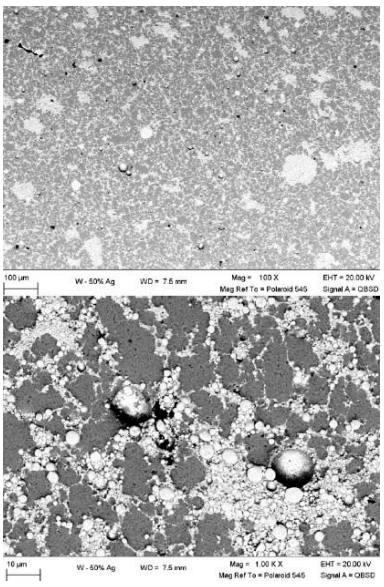


Figure 4. Microstructures of W–50%Ag HEC sample obtained at 1050 °C temperature with intensity of loading 10 GPa. (a) A large area of compositional segregation is visible at the centre of sample. This area is W rich and shows in light grey colour. Numerous cracks are also visible. (b) Distribution of Ag particles (in dark colour) – good bonding between W and Ag particles.

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As it is seen from microstructure the increase in of silver content in W–Ag composition has substantial influence on quality of HEC samples. Under similar experimental conditions, the obtained billets are characterized with compositional segregation accompanied with intensive cracking in the central part of sample.

The formation of tungsten rich central zone that is different from other parts may be explained by assuming transportation of heavy W phase by shock wave front during of movement to central part during the explosive compression. As for the observed intensive cracking in the same central region, it must be result of “Makh’s” steam effect. The intensive collision of shock waves in central part of billets causes an increase of the internal temperature and thermal stresses during the rapid cooling results in formationof intensive cracking.

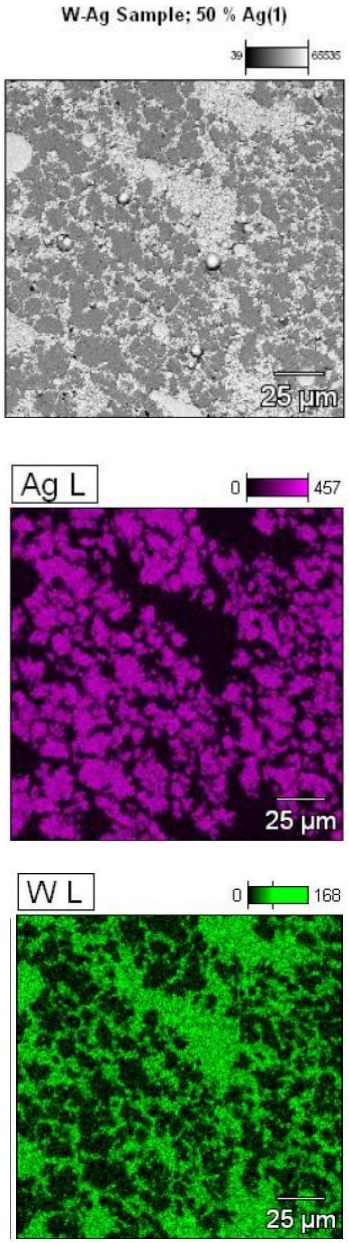

Figure 5. The location and distribution Ag (lavender colour) and W (green colour) in the matrix (grey colour) is shown. The Ag powder, initially of 8 – 10µm in size, agglomerates into nodules of average size of approximately15 to 30µm .

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The Figure 5 illustrates compositional mapping of HEC W–50%Ag sample presented in Figure 1(a).

In the W-50%Ag mixture, the combining phases show that the Ag phase, which is 8-10 μm in size at the outset, agglomerates into nodules that are about 15 to 30 εm in average size. The W-10%Ag instance also showed similar results.

A uniform heat dissipation route would be severely compromised if the W-10%Ag samples included several fractures. Electronic parts and assemblies are vulnerable to hot spots caused by uneven heat dissipation. Disparities in heat conduction and dissipation may also result from the W-50%Ag sample's W-rich central region.

In order to successfully use W-Ag composites in current microelectronics, it is important to fabricate these composites without cracks, with a density close to theoretical, excellent bonding, and a uniform distribution of comprising phases.

Under conditions of loading intensity up to 10 GPa and silver's melting point at 940 °C, the HEC was carried out to analyse the impact of temperature on the consolidation process of W-Ag precursors and to mitigate thermal stresses and cracking that occur during this process. Figure 6 shows the microstructures of several components of the final W-10%Ag concentrate.

According to the microstructures, there are no signs of compositional segregation and the whole volume of the HEC sample is devoid of fractures. Higher magnification may reveal the agglomerated, distinct W particles with interior microcracks, which may be the consequence of poor size matching and mixing between the W and Ag phases.

You can see the results of the hardness tests on the HEC W-Ag samples in Table 1, which vary with the consolidation circumstances.

Table 1. The value of hardness for HEC W–Ag composites depending on consolidation temperature obtained at intensity of loading 10 GPa.

Compacted Composites	Temp. of compaction, °C	Loading, g	Microhardness HV, kg mm^{-2}
W–5%Ag	1000	50	331.5
W–10%Ag	940	50	248.5
W–10%Ag	1050	50	208.2
W–50%Ag	1050	50	47.5

As it is seen from table the content of silver phase in composition influences the value of hardness and sample with 5 % Ag content has the best value of hardness at 331 HV. The high consolidating temperature with increased Ag content in composition leads to reduced hardness value, it might be result of intensive cracking and compositional segregation. As stated above W–Ag materials could be used for heat dissipation from electronic devices that are prone to failure at high operating temperatures. Currently, materials such as copper–tungsten (Cu–W) and aluminum–silicon carbide (AlSiC) are used in industry for heat dissipation.

The purpose of the aforementioned characterizations was to ascertain whether or not explosive consolidation produced satisfactory homogeneity, strong bonding among the metal particles, and the lack of voids. You can tell how consistent the mechanical and thermal characteristics are by looking at these microstructural traits.

The microstructures of various sections of the W-10%Ag HEC sample were obtained at a

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temperature of 940 °C with a loading intensity below 10 GPa, as shown in Figure 6. The whole billet volume is devoid of fractures, and strong bonding between the W and Ag was noted.

In order to find out whether the tungsten-silver (W-Ag) composites might be used in an electronic assembly that required heat dissipation, we tested their compatibility with other materials. These materials may include ceramics and semiconductors. Thermomechanical analysis (TMA) was used to quantify CTE, with three cycles of heat, cool, and heat, from -60 to 350 °C with a rate of 10 °C min-1. The temperature was heated twice: first from -60 to 20 °C and again from 50 to 350 °C. We average the CTE from both heating cycles for each batch of material. Table 2 below displays these values.

Table 2. Measurements of coefficient of thermal expansion, μm/m °C.

Composition	Coefficient of thermal expansion	
	–60 to 20 °C	50 to 350 °C
W–10%Ag	5.5	7.5
W–50%Ag	9.5	13.7

Conclusions

Both of the W-Ag samples that were consolidated above the melting point of silver showed signs of cracking. Severe cracking might affect the sample's mechanical integrity and heat dissipation uniformity. Hot spots, caused by a lack of mechanical integrity and non-uniformity, might harm electronics that rely on heat dissipation in real-world applications.

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Table 2 shows that when compared to other materials utilised in industry for this purpose, such as Cu-W and AlSiC, the CTE of the W-Ag samples is rather good. The Cu-W CTE values that have been reported are 6.5 for W-10%Cu and 8.3 for W-20%Cu. The W-10%Ag's CTE

the range falls under this category is 5.5 to 7.5 (µm m°C-1 or ppm K-1). As a function of composition ratio, the CTE values of AlSiC vary between 8.0 and 15 (ppm K-1). Depending on the Cu to Mo ratio, Cu-Mo (a material with high thermal conductivity) may have a CTE ranging from 7.2 to 11 (ppm K-1). The observed CTE of the W-50%Ag sample falls within this range, which is 9.5 to 13.7 (µm m°C-1). A low temperature co-fired ceramic has a CTE ranging from 4.4 to 6.4 (µm m°C-1), which was studied under same circumstances as the W-Ag samples.

The W-50%Ag sample showed compositional segregation in its central region. Around the longitudinal axis, a wide region abundant with W is seen. Because of this separation, we wonder whether the sample's characteristics, including thermal conductivity, are consistent.

Although it was outside the scope of this first assessment to evaluate the samples' thermal conductivity, it was determined that the samples' existence of several fractures would prevent them from dissipating heat evenly. Electronic parts and assemblies are vulnerable to hot spots caused by uneven heat dissipation. Disparities in heat conduction and dissipation may also result from the W-50%Ag sample's W-rich central region.

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