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Effect of Applying Hot Isostatic Pressing on the Microstructure and Mechanical Properties of Tungsten Heavy Alloys

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Abstract: The objective of this experimental study is to investigate the effect of hot isostatic pressing (HIP) on the density, mechanical properties and microstructure of pre-sintered specimens of 93%W-4.9%Ni-2.1%Fe alloy. To achieve this target, Containerless HIPing at temperature 1300°C was applied. While, the HIPing pressure was varied from 100 MPa to 150 MPa.

Elemental powders were mixed using double cone mixer for 2 hours. Cold Isostatic Pressing was applied for consolidation of metal powders into green compacts to obtain cylindrical tensile and impact specimens at 200MPa using rubber molds. Finally, the specimens were sintered under vacuum atmosphere at 1470ºC for 30 minutes. The applied HIPing temperature was chosen to be 1300°C. While, the applied HIPing pressure was varied from 100MPa to 150MPa, in order to study the effect of HIPing pressure on the mechanical properties, and the soaking time was 60 minutes.

The effect of applying Hot Isostatic Pressing was characterized in terms of density, hardness, impact resistance and tensile properties, then compared with samples in the as sintered state.

The microstructure analysis indicates that connectivity, contiguity and average grain size increase, while micropores were almost eliminated, this took place when applying a HIPing cycle at 1300°C under 100MPa for 60 minutes. On the other hand, When the HIPing pressure was increased to 150 MPa, a severe plastic deformation of the tungsten grains takes place, contiguity seriously increases leading to inhomogeneous microstructure.

The tensile strength and hardness increase when applying hot isostatic pressing of 100MPa, compared with its value in the as sintered state, Further increase in HIPing pressure up to 150MPa decreases theses values of strength and hardness, relative to its value at 100MPa. On the contrary, ductility and impact resistance decrease continuously when applying a HIPing cycles under 100MPa and 150MPa respectively, relative to its value in the as sintered state.

Keywords: Tungsten heavy alloy, Hot Isostatic Pressing, containerless HIPing.

1- Introduction

Hot Isostatic Pressing (HIP) involves the simultaneous application of isostatic pressure and elevated temperature to a workpiece in a specially constructed vessel, which results in the workpiece becoming consolidated [1]. The pressure medium used is an inert gas such as argon or nitrogen, which is pumped into a pressure vessel and pressurized up to the maximum pressure, while a furnace in the vessel produces the prescribed temperature, under these

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conditions of heat and pressure, internal pores or defects within a solid body collapse and weld up [2]. One of the major purposes of the HIP process is the post densification of the sintered materials for eliminating the micro and macro-porosity to obtain a dense and homogeneous microstructure.

This workpiece can be in the form of powder, pre-sintered compact or cast product. Only when the workpiece is in the form of powder, it is encapsulated in an evacuated capsule of sheet metal, ceramic or glass. Care must be taken in the design of the capsule and during the filling operation, to avoid distortion under compression. In the case of castings, the surface of the workpiece serves as its own capsule; encapsulation is therefore not required [1,2].

HIPing is usually conducted at a temperature greater than 0.7 T_m (where T_m is the melting point of the material). Some materials may contain a relatively low melting point constituent which aids pore removal if the HIPing is carried out between the melting point of this constituent and that of the matrix. The relatively high temperatures during HIPing are necessary to lower the yield strength and to raise the diffusivities in the material sufficiently for pore closure to occur in a reasonable time [2].

The major effect of HIPing on microstructure is the removal of porosity. However, the possible occurrence of secondary effects such as grain growth, changes in precipitate distributions and changes in segregation patterns must be considered. All these processes involve diffusion and are therefore enhanced at high temperatures. In addition, the high pressures might influence phase transformations, change melting points and crack brittle particles[2].

The pressures involved in HIPing tend to be too low in themselves to cause phase transformations. For materials which shrink on solidification, pressure will raise the melting point. This effect is small, a few degrees Celsius at most [2]. HIPing pressures can also crack those brittle particles associated with porosity in ductile matrices. Such effects increase the need for carful control of pressure and temperature ramp rates.

The HIPing process was initially developed as a means of diffusion bonding nuclear reactive components and for removal of porosity in hard materials [1]. However, its potential in a number of unrelated areas was recognized quickly, and it was applied subsequently to the major areas [3] as removal of internal casting defects, consolidation of metal and ceramic powders; either using containers filled with degassed powder, or HIPing previously sintered metallic or ceramic material without a canister. Also, HIPing has been used to bond metal-matrix composites [1], Bonding using HIP represents great advantage, where many similar and dissimilar materials can be joined; which cannot be joined by traditional fusion methods such as welding.

There is almost very little information till now about the HIPing of heavy metals available in the open literature. Danninger et al. [4] applied containerless HIPing by applying hot isostatic pressing for previously sintered alloy at temperatures between 1200°C and 1500°C to reduce porosity and improve tensile properties, two types of WHA samples have been investigated, the heavy metals with a binder phase of Cu, Ni. [4].

Optimal sintered qualities of W-Fe/Ni heavy metals could not be improved further by HIPing. However W-Fe/Ni heavy metals with lower initial tensile values could be improved by HIPing. While, the mechanical property data of W-Cu/Ni heavy metal samples could be increased by HIPing as well. Optimum HIPing temperatures for W-Ni/Fe and W-Ni/Cu heavy

metal samples were found to be around 1300°C - 1400°C. At highest HIPing-temperatures property values decreased again [4].

They reported that the W/Cu-Ni heavy metal samples show generally an increase in density by HIPing. The highest densities are obtained at 1300°C HIPing temperature. The W/Fe-Ni heavy metal sample with high initial mechanical properties shows no further increase in density. The density of this alloy remains approximately at the same level [4].

Hardness levels of all of the alloys have been recorded but no apparent trend could be found. They remain at about the same level at all HIPing temperatures without any major change. W/Fe-Ni heavy metal samples with high strength and elongation levels can be obtained with more ease than in the case of W/Ni-Cu alloys. Elongation shows a steady decrease with increasing HIPing temperature while tensile strength remains at approximately the same level.

0.Botstein studied solid phase sintering of 90W-7Ni-3Fe alloy by hot isostatic pressing (HIP) [5]. The influence of the HIP parameters (T, t, P) on the microstructure, fracture surface, and mechanical properties was investigated. The stainless steel HIP containers were filled with compacts and degassed while heated to 500-600°C. After degassing the HIP containers were closed by welding. HIP was then applied in the temperature range 1250-1350°C for a holding time of 2-6 hours, a pressure of 108 MPa being maintained. The study concluded that [5] moderate W grain size growth was observed after HIP in the temperature range 1250-1300°C (4-6h), Also, the maximum transverse rupture strength (1800MPa) was obtained after pre-sintering at 1430°C (HIP 1250°C - 2h). The prolonged HIP treatment (up to 6 hours) after pre-sintering at 950°C increased the values due to the complete closure of pores. Directly HIPed specimens, i.e., without any pre-sintering treatment, exhibited the worst properties.

G.Petzow et al. [6] studied the mechanisms for elimination of pores during HIPing. In polyphase materials consisting of a comparatively mobile intergranular phase and comparatively immobile grains, such as heavy metal alloys, large pores are eliminated during final stage HIPing by liquid flow or by a mixed liquid/grain flow. Pore filling by the mobile matrix alone results in soft spots in the material and inhomogeneous microstructure, therefore, it is undesired. Pore filling by a simultaneous flow of grains and intergranular matrix phase, is obtained by changes in the viscosity, volume fraction of intergranular liquid phase and the grain size of the solid constituents by the application of different HIP conditions.

2. Experimental Procedure

2.1 Characteristics of Used Powders

The main constituents of the adopted tungsten heavy metal alloy are commercial pure tungsten, nickel and iron powders. Tungsten powder was fabricatred by reducing tungsten oxide in hydrogen atmosphere, while, Nickel and Iron powders were fabricated by carbonel method.

The different powders were chemically analyzed by X-ray fluorescence (XRF) technique to determine their composition and purity. The obtained chemical compositions are illustrated in Table (1).

The apparent densities of these powders were measured by Hall flowmeter, and the tap densities were determined after standard tapping. The results of both densities were compared with the values of the theoretical densities as shown in Table (2).

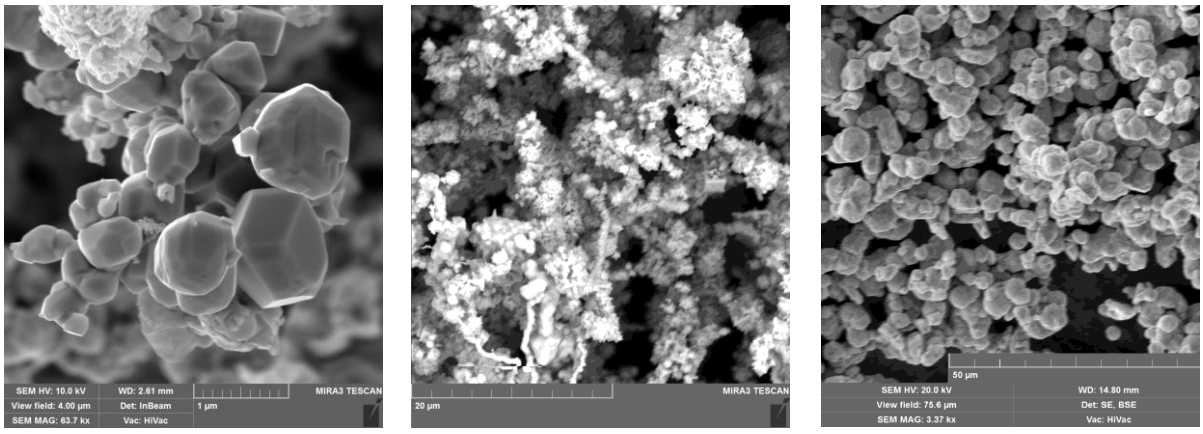
Table 1. Chemical composition of the used powders (in wt.%)

Composition / Powders	W%	Ni%	Fe%	Si%	Al%	Mg%	Ca%	Ga%	P%	S%
Tungsten powder	99.825	--	--	--	--	--	0.036	0.021	0.062	0.056
Nickel powder	--	99.496	0.132	0.204	0.115	0.046	--	--	0.003	0.003
Iron powder	--	--	99.396	0.335	0.172	0.062	0.02	--	0.01	0.005

Table 2. Theoritical, apparent and tap densities of the used powders

Powders / Measured density	Tungsten powder	Nickel powder	Iron powder
Theoretical density, g/cc	19.3	8.9	7.9
Apparent density, g/cc	3.6	0.93	1.4
Tap density, g/cc	6.75	1.65	2.63

The morphology of these powders was revealed by SEM as shown in Fig.1(a-c). Tungsten powder appered to have polygonal shape, and nickel powder is of spongy shape, while the iron powder shows nearly spherical shape. It can be noted that Tungsten, Nickel, and Iron powders have an average particle size of about 1-2 µm, 1-3 µm, and 3-5 µm respectivily.



(a) *(b)* *(c)*

Fig. (1). SEM images of as received (a) Tungsten, (b) Nickel, and (c) Iron powders

2.2 Preparation of Sintered Specimens

For improving the mechanical properties, eliminating micro and macro-porosity, obtaining a dense and homogeneous microstructure, and reducing the scatter band of properties, hot isostatic pressing process is applied, as a post sintering process for tungsten heavy alloy.

For this objective, containerless HIPing of presintered tensile and impact test specimens is performed. Samples having the chemical composition 93%W-4.9%Ni-2.1%Fe were prepared by mixing, degassing and compaction, using a rubber mold, in a cold isostatic press up to a maximum pressure of 200 MPa, followed by sintering in a vacuum atmosphere, under a temperature of 1470°C for a period of 30 minutes, then allowed to be furnace cooled to room temperature.

The optimum HIPing temperatures for W-Ni/Fe heavy metal samples were found to be around 1300°C-1400°C [4]. In our case, the applied HIPing temperature was chosen to be 1300°C, to avoid excessive grain growth. The applied HIPing pressure was varied from 100MPa to

150MPa. During the HIPing cycle, temperature and pressure were increased simultaneously to reduce the processing time, and the soaking time was 60 minutes. The applied cycles are shown in Fig.(2) and Fig.(3).

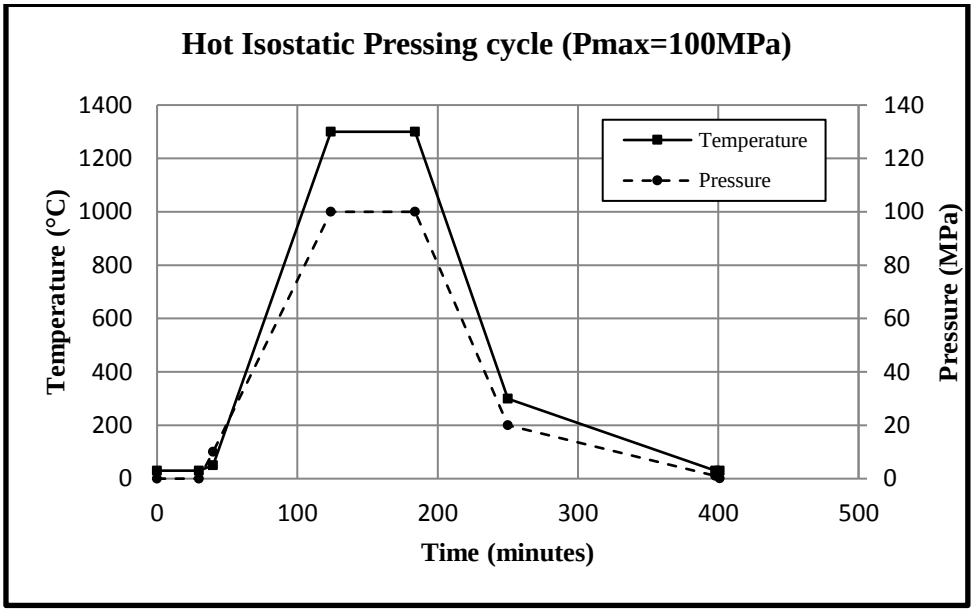


Fig.(2). HIP cycle applied for maximum pressure of 100 MPa.

The first cycle shown in figure (2) starts with an autoburge step, which takes about 30 minutes. The autoburge begins with evacuating the vessel using a rotary evacuation pump connected to the HIP vessel down to a pressure of 1 torr to get rid of any residual gases from previous cycles. Then argon gas is pumped to the vessel from the auxiliary gas system to a pressure of 1 bar, then evacuated again. This evacuation and pumping cycle is repeated twice to make sure that the vessel is clean.

After this step, the temperature is raised, firstly with a low heating rate, simultaneously the system starts pumping argon to the vessel. The pumping of the gas to the vessel is done without the compressor in a first stage. Then the compressor starts working when the pressure reaches 6.8MPa. The heating rate is increased to 15°C/min and the pumping continues till the maximum temperature 1300°C and maximum pressure 100MPa is attained. The gas is pressurized partly by its heating effect and partly by the compressor. When the prescribed values of pressure and temperature are attained, the dwell time starts for 60 minutes to allow enough densification to occur.

At the end of the soak time, the cooling stage begins, where the temperature is reduced with a high rate in a first step, till the temperature reaches 300°C. Then, the cooling rate is decreased as the actual decrease in furnace temperature becomes slow. Also, this guarantee that the actual furnace temperature and the set point programmed temperature remains matching together. Cooling the vessel continues till the temperature reaches the room temperature.

Concerning the depressurization stage of the pressed gas, the depressurization rate is adjusted to ensure that pressed gas remains during the whole cooling stage. Actually, the cooling of the vessel is done by the simultaneous effect of the running cold water, pumped by the cooling system inside a water cooling jacket, and the release of the pressed gas inside the vessel, which reduces the temperature effectively. The gas is reclaimed from the vessel back to bottles till certain pressure (about 8MPa), this reclaimed gas is recommended to be used only for another cycle to avoid the effect of contaminants.

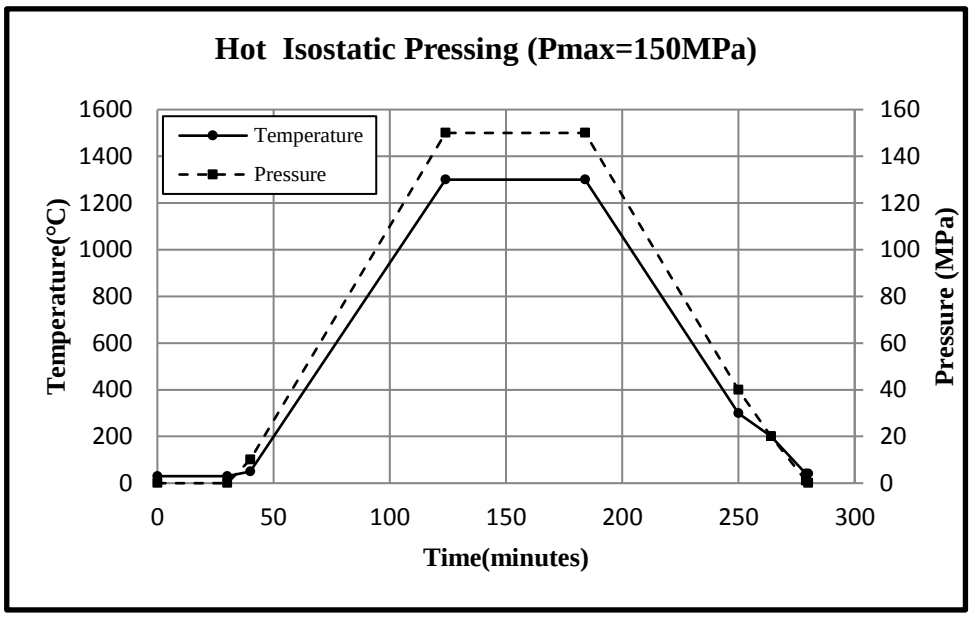


Fig.(3). HIP cycle applied for maximum pressure of 150 MPa .

The second cycle shown in figure (3) passes through the same stages as the first cycle, except that the pressure reaches 150 MPa. Tensile and impact samples, shown in figure (4), HIPed at 100MPa and 150 MPa were tested in an identical procedure, and compared to other samples tested in the as sintered state.

Fig.(4). The produced specimens used in determination of tensile strength, impact and sintered density after applying a HIPing cycle at 1300°C with different isostatic pressures.

2.3 Characterization of Samples

The densities of the sintered specimens were measured by the Archimedes water immersion method [7]. Quasi-static tensile testing was carried out using an Instron testing machine model 8032, under a load control mode of 0.15 KN/Sec. The stress–strain diagram was recorded on standard test specimens, prepared according to the ASTM standard E8M. Charpy impact test was conducted using unnotched standard impact specimens according to ASTM standard E23. The energy absorbed by specimens per unit area was taken as a measure of the impact resistance. The hardness of the produced specimens under different conditions was measured by Vickers hardness tester type Instron Wilson-Wolpert model Tukon 2100B, using 30 Kg load. Vickers micro-hardness measurements were carried out applying a load of 1000 gm, using a Vickers micro-hardness testing machine type Buhler. An average of three hardness readings, for each specimen, was determined.

Samples were prepared for microstructural evaluation by cutting, mounting, grinding and polishing to a 0.3µm surface finish using standard metallographic procedures. The microstructures were observed by scanning electron microscope (SEM) type TESCAN after etching using (HNO_3 15 ml, HF 3 ml, H_2O 80 ml) as an etchant [8]. The micrographs were quantitatively analyzed using an image analyzer to measure the size and volume fraction of tungsten particles, the matrix fraction, the connectivity and the contiguity, which greatly influence the properties of tungsten heavy alloys.

3. Results and Discussion

3.1 Effect of Applying HIP on Microstructure

The microstructure of the produced specimens, in the as sintered state, and those subjected to a HIPing cycles of 1300°C at pressures of 100MPa and 150MPa, are shown in Figs.(5) to Fig.(7). In this study, micrographs using a fixed magnification of 1200X were taken from each sample, at different locations for analysis.

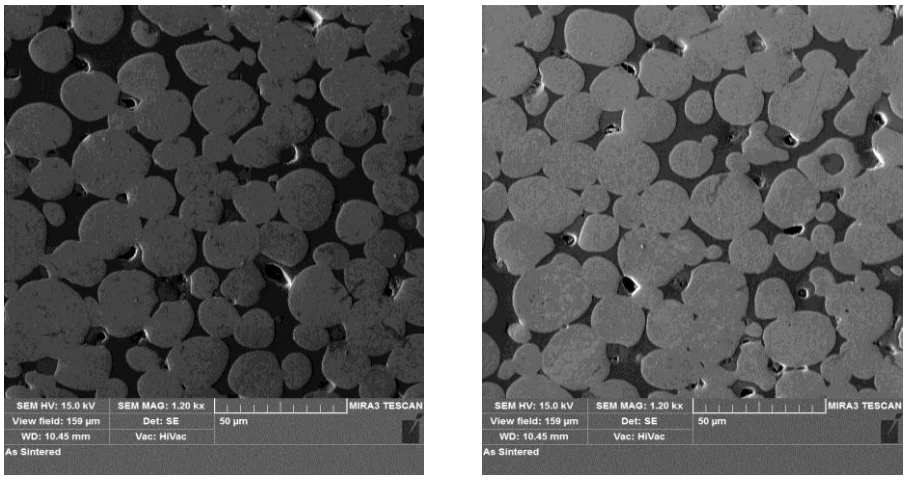


Fig.(5). SEM micrographs of the alloy 93%W-4.9%Ni-2.1%Fe CIPed at 200 MPa and sintered for 30 minute at 1470°C, using magnification 1200X.

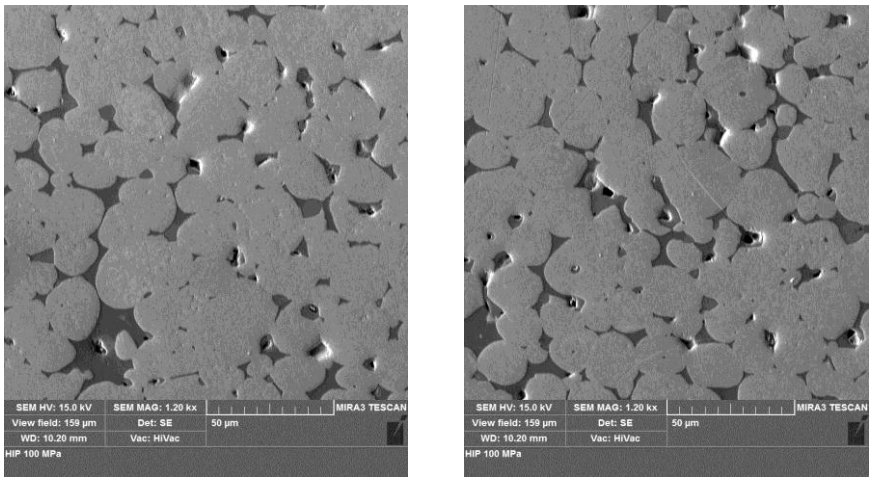


Fig.(6). SEM micrographs of the alloy 93%W-4.9%Ni-2.1%Fe CIPed at 200 MPa and sintered for 30 minutes at 1470°C, then HIPed at 1300°C under 100MPa for 60 minutes using magnification 1200X.

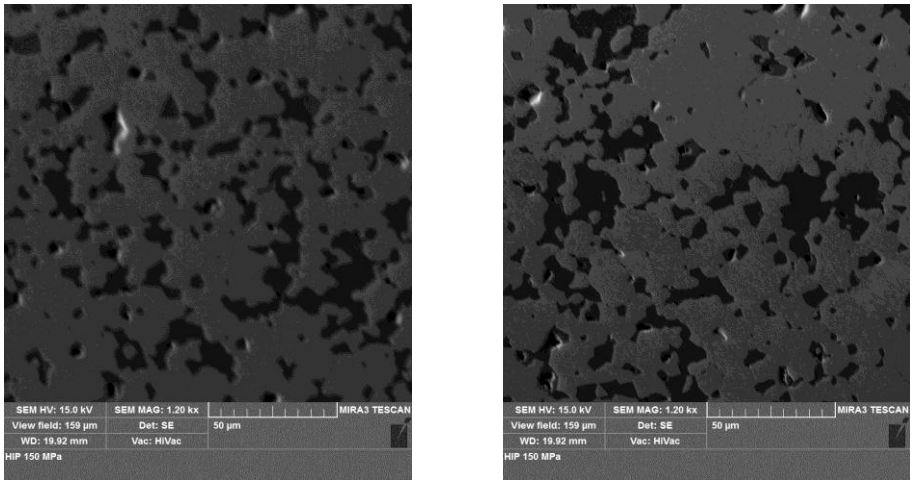


Fig.(7). SEM micrographs of the alloy 93%W-4.9%Ni-2.1%Fe CIPed at 200 MPa and sintered for 30 minutes at 1470°C, then HIPed at 1300°C under 150MPa for 60minutes using magnification 1200X.

The micrographs of the alloy 93%W-4.9%Ni-2.1%Fe CIPed at 200 MPa and sintered for 30 minutes at 1470°C revealed a uniform distribution of the matrix phase surrounding and encircling the tungsten grains. It can be also noted that the morphology of the grains is still nearly rounded with an average grain size of 18 µm as shown in Fig.(5).

The examination of the micrographs illustrated in Fig.(6) of the same alloy subjected to an extra HIPing cycle at 1300°C under 100MPa for 60 minutes showed that the distance between the centers of any two neighboring grains became shorter. Moreover, the grains became more deformed and deviate from the spherical morphology. Connectivity and contiguity are clearly increased and the average grain size is increased to about 22 µm. On the other hand, we can remark that the micropores were eliminated while the relatively larger pores became smaller in size due to the simultaneous effect of HIPing pressure and temperature. Consequently, the matrix phase became not perfectly surrounding the tungsten grains resulting in lower wetting effect of these grains.

When the HIPing pressure was increased to 150 MPa, keeping all other parameters constant, a profound evolution of the obtained microstructure can be observed as shown in Fig.(7), where we can note a severe plastic deformation and welding of the tungsten grains. Contiguity seriously increased and approaches 100%, while a pronounced redistribution of the matrix phase can be clearly seen, where the matrix phase is collected in the form of separate islands instead of being surrounding the tungsten grains leading to weak binding effect and inhomogeneous microstructure.

3.2 Effect of Applying HIP on Mechanical Properties

The stress strain curves of the alloy 93%W-4.9%Ni-2.1%Fe in the as sintered state and after applying a HIPing cycle at 1300°C for 60minutes under an isostatic pressure of 100MPa and 150MPa are shown in Fig.(8).

The effect of HIPing pressure on the ultimate tensile strength and ductility can be summarized in the bar charts illustrated in figure (9) and figure (10).

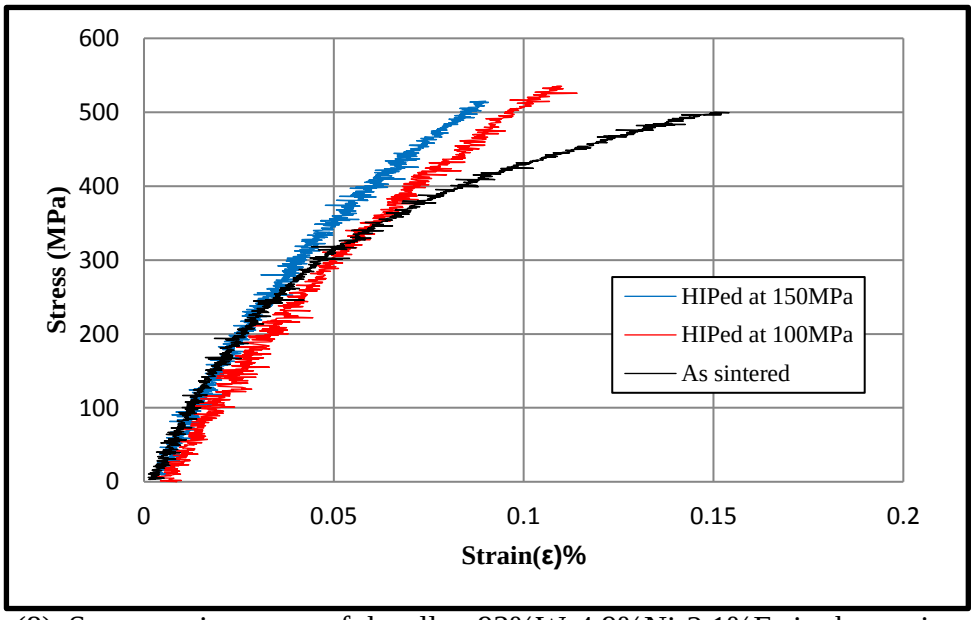


Fig.(8). Stress strain curves of the alloy 93%W-4.9%Ni-2.1%Fe in the as sintered state and after applying a HIPing cycle at 1300°C for 60minutes under an isostatic pressure of 100MPa and 150MPa.

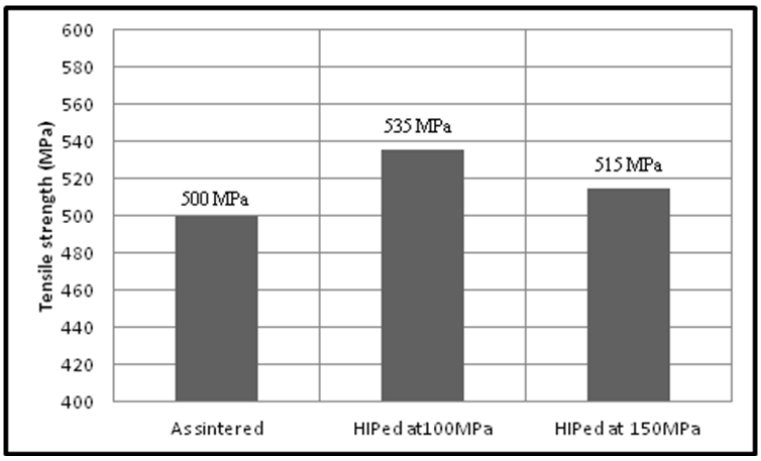


Fig.(9). Effect of HIPing pressure on the tensile strength of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60 minutes.

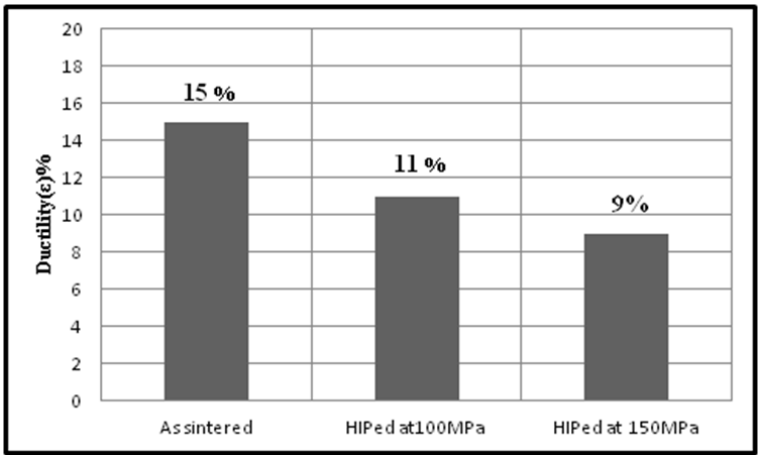


Fig.(10). Effect of HIPing pressure on the ductility of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60minutes.

It can be noted that, the tensile strength is increased with about 7% compared with its value in the as sintered state, when applying hot isostatic pressing of 100MPa. Further increase in HIPing pressure up to 150MPa decreases this strength by about 4% relative to its value at 100MPa. On the contrary, ductility decreases continiously by about 26% and 40% when applying a HIPing cycles under 100MPa and 150MPa respectively, relative to its value in the as sintered state.

In reality, the simultaneous application of high temperature and isostatic pressure during the HIPing process results in considerable mobility of either the matrix phase alone or both matrix phase and tungsten grains. This mobility, depending on the HIPing parameters, leads, from one hand to the closure of the residual pores, and from the other hand, to strain harden the tungsten phase, thus contributing to the densification and strengthening of the alloy. Excessive augmentation of the HIPing pressure results in an inhomogeneous microstructure and bad redistribution the matrix phase, which may lead to serious drop of mechanical properties. Up to a HIPing pressure of 100MPa, the mechanisms of densification and strengthening dominates those resulting in bad distribution of matrix phase. When the HIPing pressures attains 150MPa, the matrix phase became no longer surrounding the tungsten grains causing insufficient wetting, also it agglomerates in the form of separate islands and loses its binding effect, thus mechanical properties were decreased.

Fig.(11) illustrates the influence of HIPing pressure on the impact resistance of the adopted alloy. It can be noted that, the impact resistance shows a similar behaviour to ductility, as it continuously decreases, when increasing the HIPing pressure applyed.

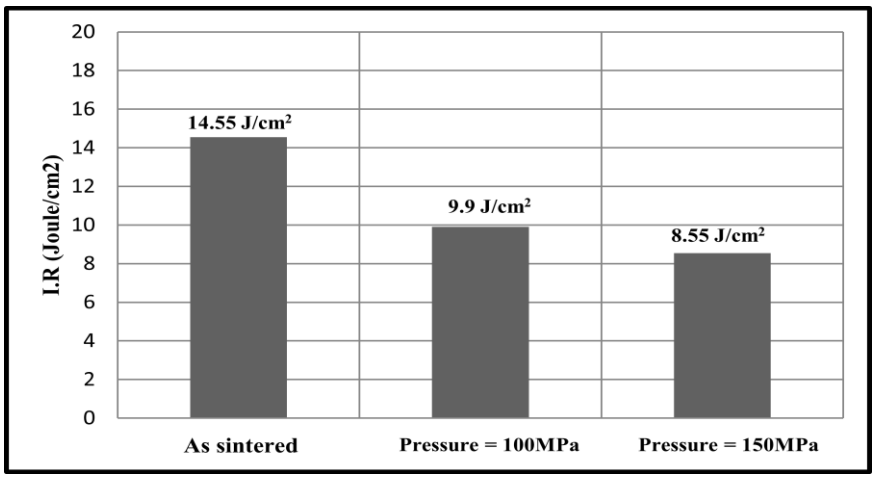


Fig.(11). Effect of HIPing pressure on the impact energy of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60 minutes.

The results of the measured bulk hardness on the different samples indicated that hardness increases by about 6% when applying HIPing at a pressure of 100MPa compared to hardness values of the samples in the as sintered condition as shown in figure (12). In a second stage, when the HIPing pressure is increased to 150MPa, the hardness is decreased by about 3% compared to the hardness of the samples HIPed at 100MPa. This behavior is similar to that observed for ultimate tensile strength.

The increase of hardness when the HIPing pressure was 100MPa can be attributed to the promotion of the densification processes, from one hand, by the high temperature plastic flow and, from the other hand, by the solid state diffusion under the effect of high pressure and temperature. When the HIPing pressure is increased to 150MPa, the decrease in hardness can

also be directly attributed to the loss of the binding effect of the matrix phase due to its mobility and agglomiration.

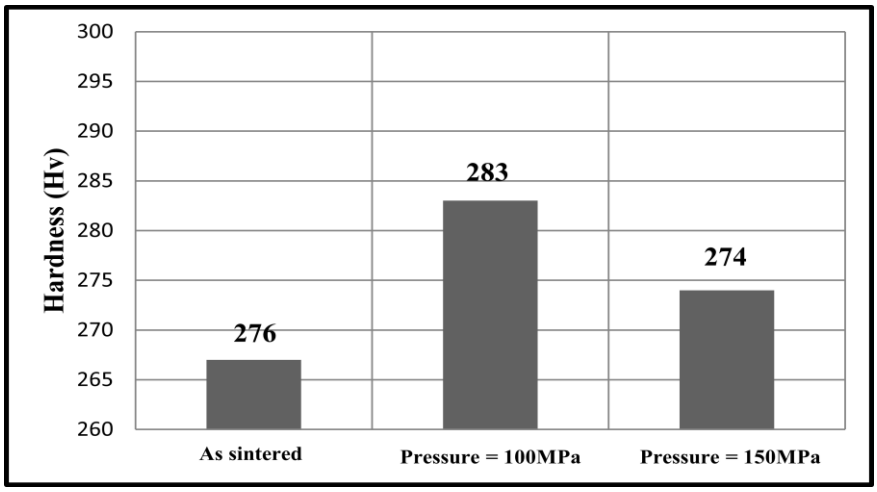


Fig.(12). Effect of HIPing pressure on the hardness of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60minutes.

To confirm the effect of HIPing on the hardness of the alloy, micro-hardness values were measured along a line through the cross section of the specimen. The results of these mesurements are illustrated in Fig.(13). It is clearly evident that the hardness increases with applying the HIP process at a pressure of 100MPa, and that the hardness on the surface was higher than the hardness at the core of the sample. With increasing the HIPing pressure to a pressure of 150MPa, the hardness values decreases as previously stated, and the difference between the hardness measured at the surface became close to the hardness at the core due to the effect of the higher pressure, which yields similar mobility of the different phases at the core and on the surface of samples. Both hardness distributions obtained after applying the HIPing cycles are higher hardness relative to the hardness distribution measured on the as sintered samples.

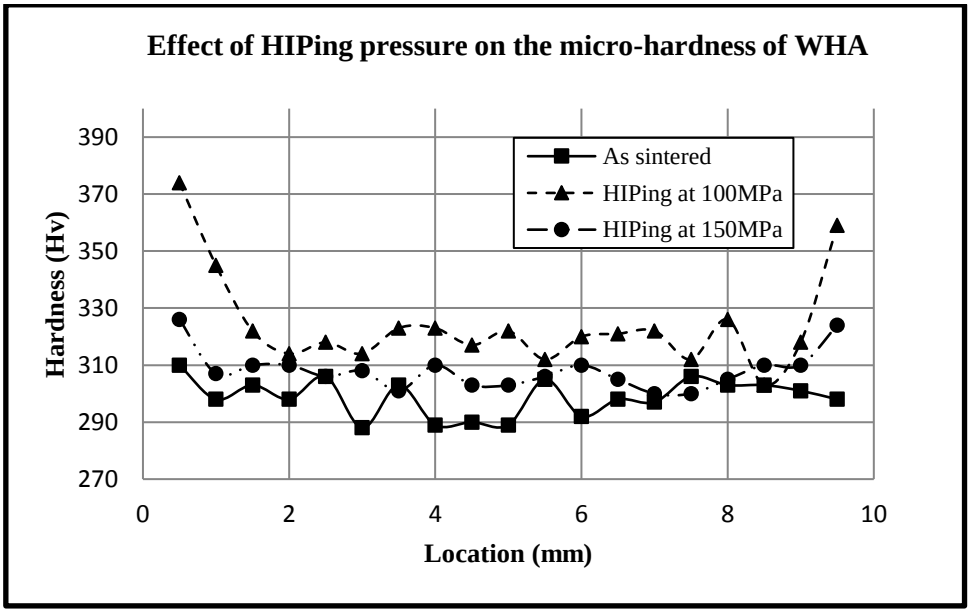


Fig.(13). Effect of HIPing pressure on the micro-hardness of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60minutes.

3.3 Effect of Applying HIP on Physical Properties

The effect of applying hot isostatic pressing using various HIPing pressures, on the sintered density of the tungsten heavy alloy, is illutrated in Fig.(14).

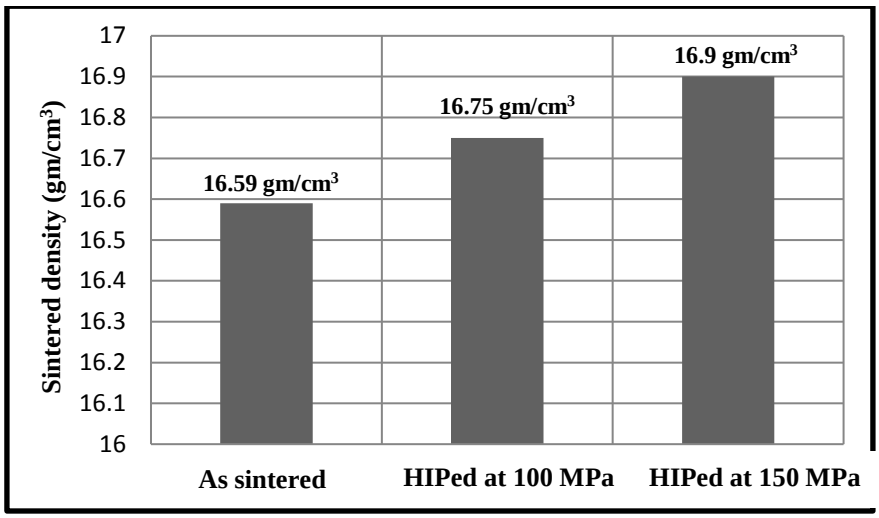


Fig.(14). Effect of HIPing pressure on the sintered density of the alloy 93%W-4.9%Ni-2.1%Fe, HIPed at 1300°C for 60minutes.

It can be noted that, applying hot isostatic pressing at pressure 100MPa causes a slight increase in the sintered density with about 1% compared to its value in the as sintered state. With increasing the HIPing pressure to 150MPa, the sintered density increased again with about 1.8% compared to its value in the as sintered state. This increase with increasing the HIPing pressure can be related to the increase of diffusional creep, and pore elimination by the mobility of the matrix phase. In fact, the sintered density, unlike the mechanical properties, is insensitive to the matrix phase distribution.

4. Conclusion

This study has determined the variations in microstructure and mechanical properties for tungsten heavy alloy with different HIPing pressures and constant HIPing temperature.

- Applying hot isostatic pressing cycle at a temperature of 1300°C under a pressure of 100MPa for 60 minutes, as a post sintering operation for the pre-sintered 93%W-4.9%Ni-2.1%Fe alloy, micropores were almost eliminated, while macropores became smaller in size. Furthermore, Connectivity, contiguity and the average grain size are clearly increased. The net increase in strength was about 7% compared with its value without applying a HIPing cycle.

- When the HIPing pressure was increased to 150 MPa, a severe plastic deformation and welding of the tungsten grains took place. On the other hand, the matrix phase was collected and redistributed in the form of separate islands leading to weak binding effect and inhomogeneous microstructure. Consequently, tensile strength, ductility, and impact resistance were significantly decreased.

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