



Effect of Cold Isostatic Pressing on the Physical and Mechanical Properties of Tungsten Heavy Alloys

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Abstract: The objective of this experimental study is to investigate the effect of cold isostatic pressing (CIP) on the density, mechanical properties and microstructure of 93%W-4.9%Ni-2.1%Fe alloy. Also, to compare between the effect of uni-axial pressing and cold isostatic pressing on the alloy properties.

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 under different pressures using rubber molds. Finally, the specimens were sintered under vacuum atmosphere at 1470°C for 90 minutes.

The results of density, tensile strength, ductility, hardness and impact resistance obtained from the produced specimens were compared with those obtained using uni-axial compaction. Moreover, the values of the isostatic compaction pressure were varied in order to study their effect on the mechanical properties and to determine the optimum value providing the highest properties.

The microstructure analysis indicates that uni-axial compaction can provide a structure with lower contiguity, and a grain size relatively course, while isostatic pressing provides a structure with fine grains and higher contiguity.

The obtained results of all measured mechanical properties prepared by cold isostatic pressing showed higher values than those measured on specimens uni-axially compacted, for all values of pressure. We can, also, notice that the relative green density is higher, in case of applying CIP technique, for all values of compaction pressure. It can be concluded that cold isostatic pressing leads to a sensible improvement in alloy properties.

Keywords: Tungsten heavy alloy, Cold Isostatic Pressing, Uni-axial Pressing.

1. Introduction

It was Blaise Pascal, the French scientist, who proposed that "pressure applied to a confined fluid at any point is transmitted undiminished through the fluid in all directions and acts upon every part of the confining vessel at right angles to its interior surfaces and equally upon equal areas" [1].

The application of Pascal's law allows powder contained in a flexible bag or envelope, to be densified under pressure acting through a suitable pressure transmitting medium. The pressure acts equally over the surface of the bag which, being flexible, squeezes the powder uniformly to a compact whose external geometry is smaller than, but of similar shape to that of the original bag, which is the principal of isostatic pressing.

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The first attempt to exploit Pascal's law was made in 1913 by Madden, who described an isostatic pressing technique to produce refractory metal filaments for electric lamps. Madden's process was designed to overcome many of the difficulties that were being met in the die compaction of fine none ductile powders such as tungsten and molybdenum. Typical of such difficulties were the incidence of cracks, laminations, non-uniform properties and lack of green strength sufficient to withstand the subsequent handling without fracture [1].

Subsequent development of CIP took place relatively slowly [1]; in 1928 Feshe described an isostatic process for the pressing of tungsten tubes. In the early 1930s, Jeffrey along with Daubenmeyer, were the first to fabricate ceramic articles isostatically. Further work on isostatic pressing developed by the later 1930s, and by the early 1940s, stimulated by the need for special metals, explosives and other materials during world war II development progressed from the laboratory into large scale production.

Further progress of CIP has been slow, particularly for metal powders, compared to a more active and commercial exploitation of the process within the ceramics field. While die compaction has remained the principal method for fabricating metal powders, it too has specific technical limitations that restrict the manufacture of more complex shapes and geometries. Hence, CIP is now being increasingly used in the powder metals field as an alternative to die compaction for the manufacture of such complex shapes.

Cold isostatic pressing is an important method of compacting powders (metallic, ceramic, cermets, chemical compounds and pharmaceutical products) into high density, near net shape green parts in a high pressure vessel. Many of the constraints that limit the geometry of parts compacted by unidirectional pressing in rigid dies are eliminated by this process. Long thin-walled cylinders and parts with undercuts can be readily fabricated.

In cold isostatic pressing, pressure is applied simultaneously and equally in all directions, using a fluid (water or oil) to an elastomeric mould filled with powder at room temperature. Sintered cold isostatically pressed component can reach around 95 to 97% of theoretical density with enhanced mechanical properties [1]. Further increase in density may be achieved by cold working the sintered part or by Hot Isostatic Pressing (HIP).

In uni-axial compaction, rigid die sets can be expensive to manufacture and are only justified for the production of parts in large quantities, where surface detail and tolerance levels are too very high standards, and which are largely independent of pressing pressures. In isostatic pressing the flexible tool set is more sensitive to pressing pressure and it is difficult to control precisely shape and dimensional tolerances on the compact surfaces which are in direct contact with the flexible tool [1]. However, one of the advantages of CIP is that only relatively simple tooling is required. In practice, the tooling may vary from a simple envelope to a more sophisticated assembly of elastomeric and rigid metal parts.

The most important requirement of the flexible envelope for CIP, is related to the loose void space normally occupied by air, between the powder particles following filling. This air is sealed within the envelope and does not escape during compaction. The reduction in volume of the powder mass means that the highly compressible air forms pockets which can form in isolated areas. These in turn, can exert internal pressures within the compact and at the compact/envelope interface. That is why, de-gassing before the application of compaction pressure is essential.

There are two major types of cold isostatic pressing techniques [2], namely the wet bag and the dry bag processes. Both the processes make use of an elastomeric mould, resembling the

final part, in which the loose powder is kept. In the wet bag process, the mould is removable. While in the dry bag process, the mould is fixed to the pressure vessel. The determination of each type and its utilization is dependent upon the nature, quantity, size and design of the end product and the degree of automation possible in the compaction process.

Wet and dry-bag CIP were compared in more detail by Jackson [3]. A potential limitation of dry-bag CIP is that the lateral pressure exerted on the powder by the mould is transmitted to the upper and lower end closures, and unless the end axial loads are managed properly, the axial pressure may be less than the lateral pressure. Control of the axial pressures can be achieved by the use of an external axial piston, as described by Koerner, [3] who called this technique tri-axial compaction.

With both types of tooling some form of external support may be necessary to prevent the mould deforming when filled with powder. Usually perforated metal supports are provided around the moulds and assist in maintaining control of shape. Using CIP, it is difficult to achieve dimensional tolerances on outside surfaces of better than $\pm 3\%$ [1]. Only by machining the surfaces of the compact in the green or the sintered state can close tolerance control be achieved.

In comparison with conventional unidirectional die compaction, CIP has the following advantages [4]:

- In most cases, higher green densities can be obtained by isostatic pressing as compared to die pressing under similar pressures or to other densification methods.
- Green densities are more uniform in all directions and are not so strongly influenced by size and shape.
- Because of the absence of friction effects, residual stresses created in compacts are lower.
- Higher green strengths of the compact are obtained. The green strengths obtained with isostatic pressing may be between 10 and 15% higher than those obtained by die pressing or other methods.
- In most cases very little or no binding / lubricant additives are required for pure compacting purposes.
- Machining of the part at the green stage is possible by almost any of the usual machining techniques: milling, sawing, turning and grinding.
- Because of the higher and regular green densities, the minimal presence of additives and absence of irregular stresses and structure layering, shrinkage during sintering is reduced, sometimes by more than 50% and much more regular. Consequently, shrinkage becomes more controlled which is important as far as tolerance control is concerned.
- No limitations exist in dimensions or in ratios between certain dimensions. Because of the isostatic pressure, an equal pressure reaches each point of the compact, and as a result, increased ratios between length and cross-sectional dimensions can be pressed.
- Because of the omnidirectional nature of the compaction, odd shapes and forms can be made, on condition that the compacted shape can be cleared from the mould.
- Costs for dies are lower: rubber type moulds, once the original form exists, can be easily reproduced and are very inexpensive compared to other types of dies.

On the other hand, the main dis-advantages for cold isostatic pressing is that it sacrifices dimensional control and pressing speed [5].

Much of the previously published work on powder consolidation has described density-pressure relationships. There have been few previous reports of the consolidation dynamics of tungsten powder. B.P.Bewlay describes the consolidation dynamics of tungsten powder

during cold isostatic pressing [6]. Tungsten powder was consolidated using pressures from 14 to 211 MPa at pressurization rates between 2 and 10 MPa/sec.

Experiments were conducted to investigate the effect of isostatic compaction pressure, pressurization rate and axial stress on the density and microstructure of the compact. The axial expansion of the compact was also measured as a function of compaction conditions. Also, the temperature history of the compact during pressing was also determined.

The compacts were pressed at a constant rate of 8 MPa/s, for pressures from 14 to 211 MPa. In the early stage of compaction, the rate of density increase with increasing pressure was very high and decreased with increasing pressure. So, the compact density is controlled principally by the pressing pressure for a given particle size distribution.

The axial relaxation that arose during decompression of compacts generated using pressures from 14 to 211 MPa was studied. The axial relaxation was defined as the increase in the length of the compact after decompression normalized with respect to the length of the mould cavity, or distance between the punches, at the compaction pressure. For compaction pressures between 14 and 68 MPa, the axial relaxation increases from 0.13 to 0.37%. However, between 68 and 211 MPa, the additional increase was only 0.18%.

Pressurization rates between 2 and 11 MPa/sec were investigated, but no effect on the compact density, compaction ratio and axial expansion were measured [6].

Although CIP is performed without externally applied heat, some heat is generated within the compact by friction between the particles as they slide over one another [6]. The actual temperature increase depends upon the rate of pressurization and the thermal characteristics of the press tooling, but increases with increasing pressurization rate. In the case of tungsten, it is unlikely that the heat generated during CIP will increase either the density or green strength of the compact [6].

Elastic spring-back or relaxation after pressing has been reported by Lloyd and Symonds and Highriter et al. [7] also recognized that when compacting tungsten on a steel former, the relaxation when the pressure was released was sufficient to rupture the compact.

In a recent work on press and sintering of nanoscale tungsten, Olevsky and German [8] have indicated that the use of high compaction pressure can help in reducing the sintering time and temperature, thus preventing grain coarsening. The smaller the particle size, higher is the required compaction pressure. This study indicates the influence of compaction pressure (or green density) on the sintering behavior.

The main parameters for the cold isostatic pressing cycle include maximum applied pressure, pressurization and decompression rates, and hold time (dwell time) at the maximum pressure. It was reported that pressurization and decompression rates and hold time have minor effects on the properties of the compact unless the compact is subjected to a sudden decompression, that may result in serious deteriorative effects [6].

The main focus of this study is to investigate and identify the effect of cold isostatic pressing (CIP) on the density, mechanical properties and microstructure of 93% W-4.9%Ni-2.1%Fe alloy. Also, to compare between the effect of uni-axial pressing and cold isostatic pressing on the alloy properties.

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 fabricated 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).

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

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

Measured density \ Powders	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.

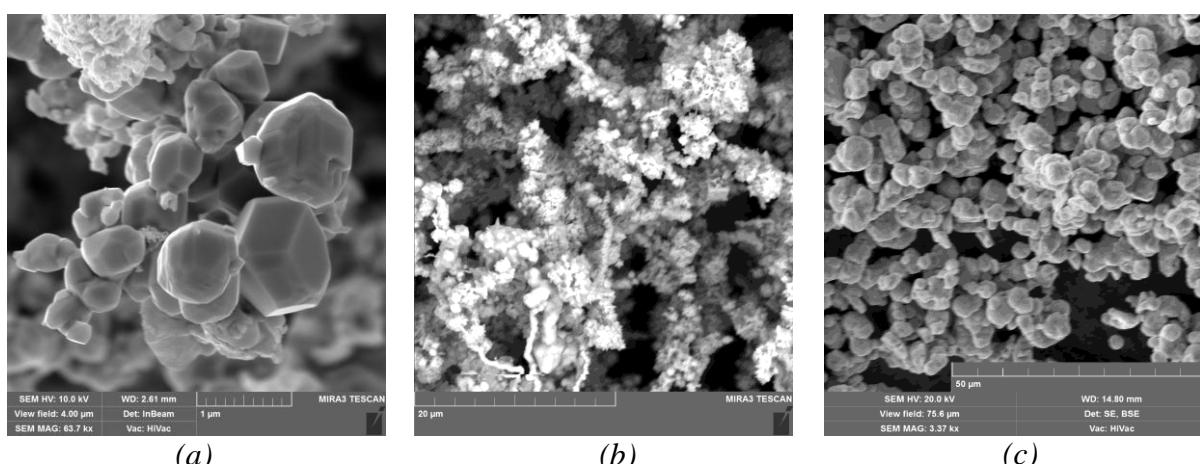


Fig. (1). SEM images of as received (a) Tungsten, (b) Nickel, and (c) Iron powders

2.2 Preparation of Sintered Specimens

The production of sintered specimens was achieved by conventional powder metallurgy technique. Elemental powders of tungsten, nickel and iron were mixed to produce a mixture with the composition of 93W–4.9Ni–2.1Fe in weight percent. This content of tungsten is widely used to achieve the highest mechanical properties, while, the Ni to Fe ratio is typically maintained to 7/3 to avoid the formation of brittle intermetallic phases [9,10,11,12]. The different powders were mixed using double cone mixer for 2 hours, to insure homogeneous mixing.

Cold Isostatic Pressing was applied for consolidation of metal powders into green compacts to obtain cylindrical tensile and impact specimens under different pressures (100, 200, 250 and 300 MPa) using rubber molds after applying degassing for 12 hours in a wet bag press, applying the pressing cycle shown in Fig. (2).

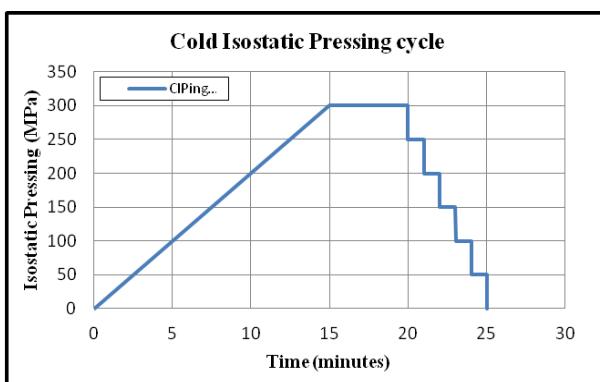


Fig.(2). Pressing cycle applied for Cold Isostatic Pressing

In this cycle, the pressurization rate is 20 MPa/min, and it is constant during the cycle, the hold time is 5 minutes. The decompression stage was divided into equal steps, while holding the pressure for a minute between each two successive steps, to avoid occurrence of cracks in the green part due to complete sudden release of pressure.

For comparison, another group of test specimens were subjected to uni-axial compaction, and shaped into standard tensile and impact specimens applying different compaction pressures. To improve compressibility during uni-axial compaction, 0.5% paraffin wax was added to the mixture for decreasing friction among particles, and Zinc sterate was used as a die wall lubricant during compaction.

Sintering was carried out under vacuum atmosphere at 1470°C for 90 minutes. The applied sintering cycle is shown in Fig.(3), it consists of the following steps:

- Heat up to 250°C at 3 °C/min and holding for 15 min. for wax removal.
- Heat up to 600°C at 6 °C/min. and holding at 600°C for 30 min. for internal stress relief.
- Heat up to 1000°C at 6 °C/min. and holding at 1000°C for 30 min. to facilitate partial solid phase sintering.
- Heat up to sintering temperature 1470°C at 6°C/min. and holding for the sintering time 90min.
- Slow cooling at 3°C/min down to 1420°C to avoid cracks during solidification shrinkage followed by furnace cooling.

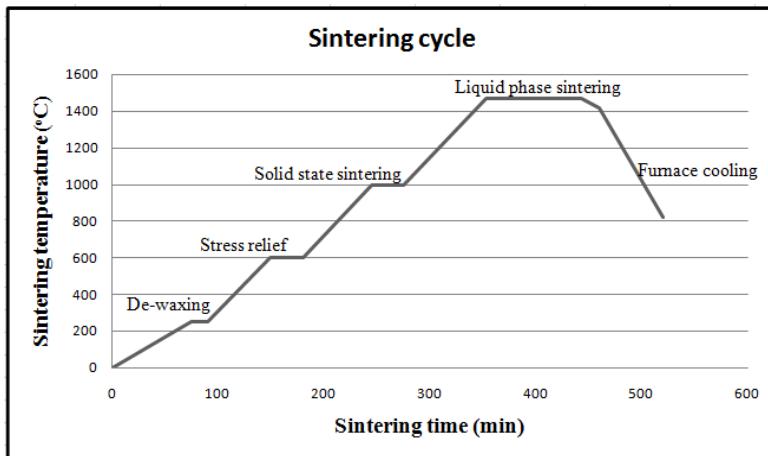


Fig. (3) Sintering cycle used for all samples.

2.3 Characterization of Samples

The densities of the sintered specimens were measured by the Archimedes water immersion method [13]. 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 un-notched 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. 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 $0.3\mu\text{m}$ 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 [12]. 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 CIP on Microstructure

Fig.(4-a) illustrates the microstructure obtained on specimens produced by traditional uni-axial compaction under 200 MPa. We can note that the contiguity[‡] is relatively low which allows the matrix phase to surround the tungsten particles. This structure and distribution of phases permit the growth of tungsten particles, during the sintering process through the major mechanism of dissolution of smaller grains and re-precipitation on the large ones.

On the other hand, Fig.(4-b) illustrates the microstructure obtained on specimens having the same composition but produced by cold isostatic pressing under the same value of pressure. We can state that, the contiguity is noticeably higher relative to that observed in the case of uni-axial compaction. However, the grain size obtained after sintering is comparatively

[‡] **Contiguity** of tungsten grains in tungsten alloy can be defined as the relative fraction of tungsten-tungsten interfacial area.

smaller. Moreover, the centers of the different grains become closer, in the different directions.

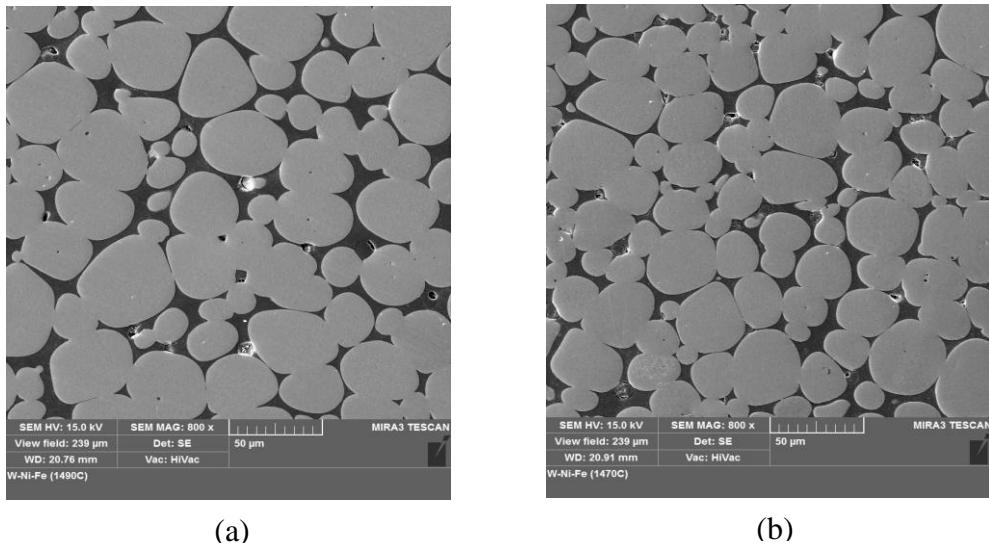


Fig.(4). Effect of compaction technique on the microstructure of tungsten heavy alloy (93% W-4.9%Ni-2.1%Fe) using the same compaction pressure of 200MPa, sintering is then carried out at a temperature of 1470°C for 90 min.
a) Uni-axial pressing, b) Cold isostatic pressing (CIP).

This evolution of the obtained structure, when applying cold isostatic pressing, is directly attributed to the uniformity of compaction in the different axes of the specimen, and the absence of friction, between the plunger or die walls and powders, which manifest in the case of uni-axial compaction. This friction affects to a great extent the densification process. On the other hand, when the contiguity is higher the contacts among tungsten particles increase, while their contacts with the matrix decrease. This effect hinders to a great extent the major dissolution and precipitation diffusion mechanism which allows obtaining relatively finer grain structure. This proves that uni-axial compaction can provide a structure with favorable lower contiguity with a grain size relatively course, while isostatic pressing provides a structure with fine grains and of higher contiguity. In fact, it can be concluded that the resulting mechanical properties using these two different compaction techniques are controlled by the effect of two factors which are contiguity and grain growth. When applying cold isostatic pressing, the increased contiguity among tungsten grains lowers the mechanical properties. However, this effect is counterbalanced by the promoted refining and densification effects, improving these properties.

3.2 Effect of Applying CIP on the Physical Properties

3.2.1 Green density

The green densities of compacts prepared by applying conventional and cold isostatic pressing were measured and their calculated relative values with respect to the theoretical density of the adopted mixture 93% W-4.9%Ni-2.1%Fe ($18.551\text{gm}/\text{cm}^3$) are shown in Fig.(5).

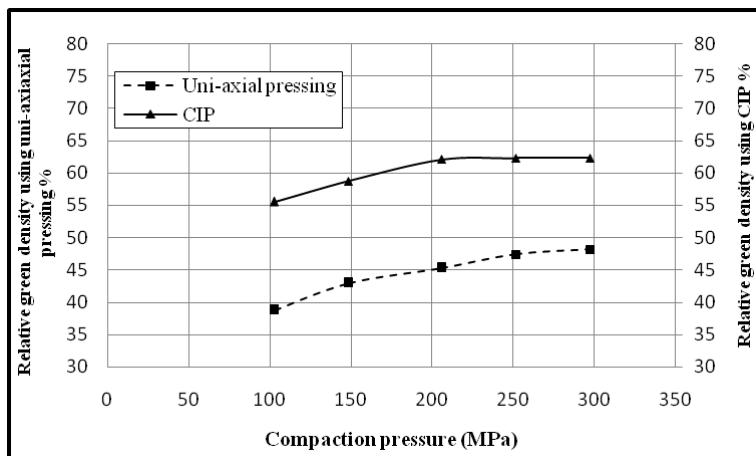


Fig.(5). Relative densities of the green compacts prepared by uni-axial pressing and cold isostatic pressing techniques.

From the figure, it is evident that, in the early stage of compaction, the rate of density increase with pressure was relatively high up to 200 MPa. Then, it decreases with further increase of pressure. We can, also, remark that the relative green density is sensibly higher, in case of applying CIP technique, for all values of compaction pressure. At a compaction pressure of 300 MPa, the relative green density attains about 49%, in case of using uni-axial pressing, while, it reaches 62.5%, in case of using cold isostatic pressing. This increase indicates that cold isostatic pressing leads to great improvement in the green density of the compact.

3.2.2 Sintered density

The relative sintered densities of the specimens produced by uni-axial pressing and cold isostatic pressing techniques under different compaction pressures (100, 200, 250 and 300 MPa) followed by sintering at a temperature of 1470°C for 90 minutes, are shown in Fig.(6). We can note that the application of cold isostatic pressing results in a slight increase of the relative sintered density of specimens relative to uni-axial pressing. This can be directly attributed to the starting higher and more uniform relative green density in case of applying CIP technique.

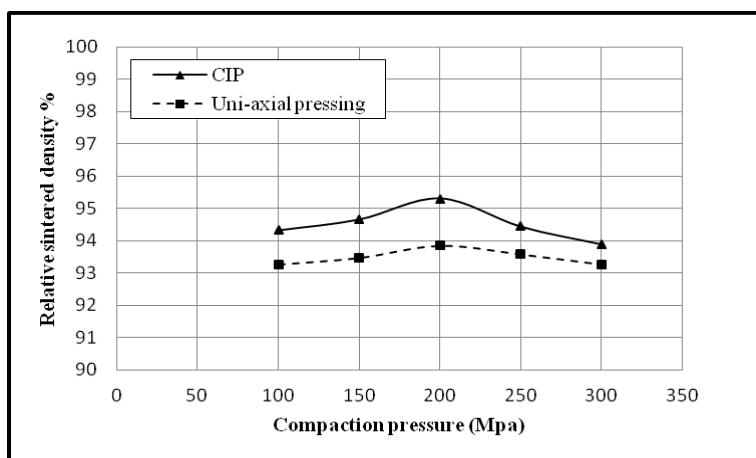


Fig.(6). Relative sintered densities of specimens prepared by uni-axial pressing and cold isostatic pressing techniques.

The measurement of sintered density of different specimens using CIP technique showed that increasing the value of cold isostatic pressure from 100 MPa up to 200 MPa, slightly, improved the relative sintered density by about 1%. When the value of cold isostatic pressure was increased above 200 MPa the density decreases, as shown in Fig.(6). This phenomenon

can be associated to the entrapped gases inside the pores during their escaping under higher compaction pressure in the green state or at elevated temperatures during sintering.

3.3 Effect of Applying CIP on the Mechanical Properties

The mechanical properties of the prescribed tungsten heavy alloy prepared by applying cold isostatic pressing technique compared to those prepared by applying uni-axial pressing technique are illustrated in Fig.(7) to Fig.(10).

Tensile strength of the sintered specimens at 1470°C for 90 minutes, prepared by uni-axial pressing and cold isostatic pressing techniques with different values of compaction pressures, is illustrated in Fig.(7). We can clearly note that, the ultimate tensile strength increases with increasing the compaction pressure up to 200 MPa, where it starts to saturate applying both compaction techniques. This strength is higher, by about 18% in case of cold isostatic pressing for all values of pressures.

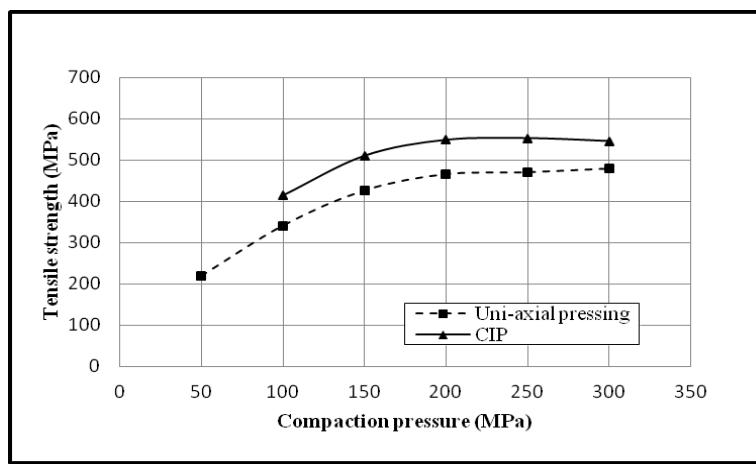


Fig.(7). Tensile strength of the sintered specimens at 1470°C for 90 min., prepared by uni-axial pressing and cold isostatic pressing techniques with different values of compaction pressure.

The measured ductility showed the same trend as ultimate tensile strength as shown in Fig.(8). This ductility is higher, by about 45% in case of cold isostatic pressing at a compaction pressure of 200 MPa.

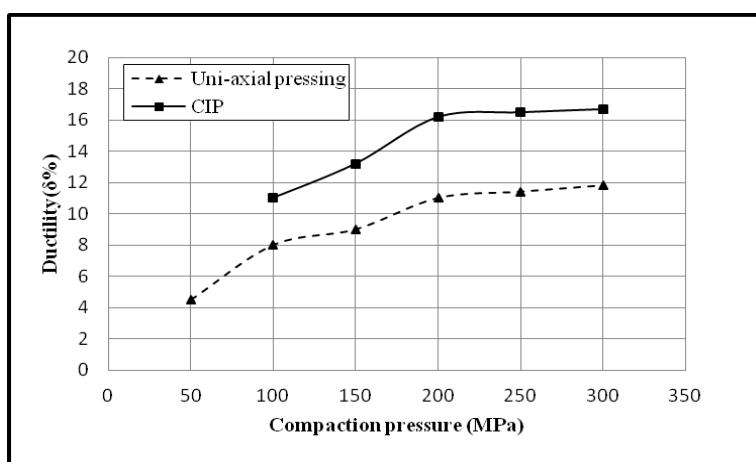


Fig.(8). Ductility of the sintered specimens at 1470°C for 90 min., prepared by uni-axial pressing and cold isostatic pressing techniques with different values of compaction pressure.

On the contrary, impact resistance of the alloy increases in a first stage, up to 200MPa, then it starts to decrease as shown in Fig.(9). Moreover, all the toughness values measured with specimens produced applying isostatic pressing are higher than those produced by uni-axial pressing, for all values of pressures. The drop in toughness at higher pressures above 200MPa can be explained, from one hand, by excessive localized plastic deformation and work hardening, causing embrittlement, which may persist even after sintering, and from the other hand, by the entrapped gases effect inside closed pores at high pressures.

The measured hardness on specimens prepared by cold isostatic pressing showed higher values than those measured on specimens uni-axially compacted, for all values of pressures, as shown in Fig.(10). Hardness showed very similar trends for the different values of compaction pressures using both techniques. In a first stage, densification is promoted, consequently hardness increases, then, in a second stage, entrapped gases inside closed pores trying to escape out, and vent towards the surface during sintering, causing vents and residual porosity, causing sensible decrease of densification and hardness.

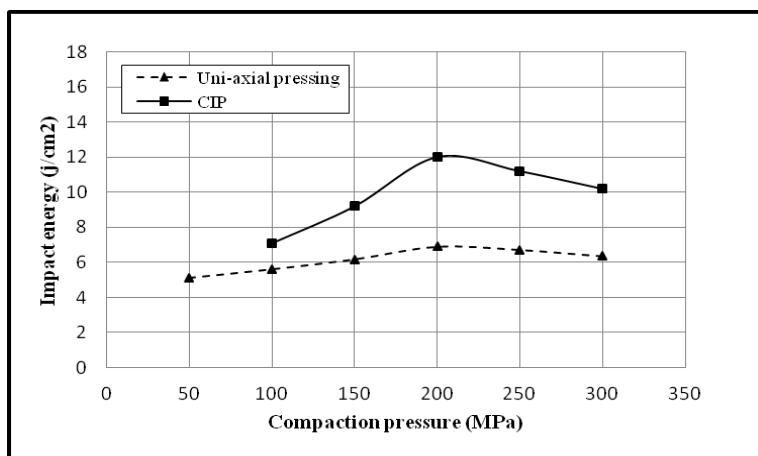


Fig.(9). Impact resistance of the sintered specimens at 1470°C for 90 min., prepared by uni-axial pressing and cold isostatic pressing techniques with different values of compaction pressure.

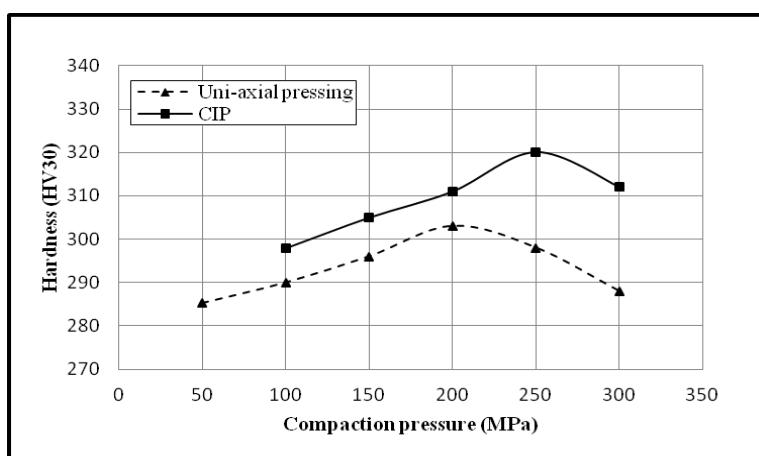


Fig.(10). Hardness of the sintered specimens at 1470°C for 90 min., prepared by uni-axial pressing and cold isostatic pressing techniques with different values of compaction pressure.

4. Conclusion

This study has determined the effect of cold isostatic pressing compared to uni-axial die pressing, on the properties of liquid phase sintered tungsten heavy alloy 93%W-4.9%Ni-2.1%Fe. This evaluation is based on the obtained microstructure and mechanical properties.

- Uni-axial compaction provides a structure with favorable lower contiguity with a grain size relatively coarse, while isostatic pressing provides a structure with fine grains and of higher contiguity.
- The application of the cold isostatic pressing raised the relative green density of compacts from 49%, in case of uni-axial pressing, to 62.5%, under a pressure of 300MPa.
- Mechanical properties for samples prepared by cold isostatic pressing showed higher values than those measured on specimens uni-axially compacted, for all values of pressure.
- Ultimate tensile strength, and ductility of the alloy 93wt.%W-4.9wt.%Ni-2.1wt.%Fe increased by about 18%, and 45%, respectively, applying cold isostatic pressing relative to uni-axial pressing under a compaction pressure of 200 MPa. While, impact resistance and hardness reaches 12 J/cm², and 320 Hv, respectively under the same pressure.

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