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Effect of Limited Additions of Y_2O_3, ZrO_2 and Al_2O_3 on the Mechanical and Microstructure Characteristics of Tungsten Heavy Alloys

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Abstract: In this experimental investigation, the effect of adding limited contents of yttrium oxide Y_2O_3 (0.02-0.5 % wt), zirconium oxide ZrO_2 (0.1-0.5% wt), and aluminum oxide Al_2O_3 (0.5-3% wt) on the mechanical and microstructure properties of the tungsten heavy alloy (90W-7Ni-3Fe %wt).Elemental powders and oxide additives were mixed for 120 min. Green compact in the form of standard tensile and impact specimens were fabricated by applying uniaxial compaction under 200 MPa. Finally the specimens were sintered at 1480ºC for 90 min under vacuum atmosphere. It was found that the addition of yttrium oxides in the order of 0.02-0.04% wt provides the highest improvements of both tensile strength and impact toughness. Moreover the addition of aluminum oxides in the order of 1% wt has strong effect in enhancing only the tensile strength, while the impact toughness is sensibly reduced. The addition of zirconium oxide in the non-stabilized form up to 0.5%wt reduces significantly the tensile properties. Furthermore, the addition of yttrium and zirconium oxides in the indicated ranges has no significant effect on the sintered density of compacts .On the other hand; the addition of aluminum oxides slightly reduces this density.

Introduction

Tungsten heavy alloys, containing from 88 to 97% wt tungsten as a base ingredient having BCC structure and a binding matrix of Ni-Fe having FCC structure are widely uses in many application like kinetic energy penetrator, air to air rocket nozzles and armor plating. That is due to their very strong inter-atomic bonding, high mechanical properties, low heat conductivity, low thermal expansion, and high corrosion resistance[1, 2].

In order to achieve as high kinetic energy as possible for kinetic energy penetrators, high density materials are required. Only there are two important candidate materials which can satisfy this high density requirement, they are depleted uranium (DU), and tungsten heavy alloys. DU alloys are currently the most widely used and favored materials for log rod kinetic energy penetrators.

This is essentially attributed to the high density, and elevated mechanical properties, in addition to the self-sharpening phenomenon that results from the adiabatic shear that generally occurs during material deformation at high strain rates[3].

Because of the increasing environmental concerns due to contamination, resulting from residual radiation, intensive efforts were done to find suitable substitutes for these alloys. In the last decades, extensive researches were carried out to improve the penetration and ballistic properties of tungsten heavy alloys to be the appropriate substitute of DU alloys.

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Tungsten heavy alloys are manufactured only by powder metallurgy technique, due to the very high melting temperature of tungsten. Tungsten heavy alloys contain W with some combinations of Ni, Fe, Co, or Cu. After mixing and compaction, these elemental powders are liquid phase sintered mostly at temperature above 1450ºC. Liquid phase sintering is usually done under hydrogen, argon or vacuum atmospheres[4, 5].

To enhance the mechanical and the ballistic characteristics of tungsten heavy alloys, there are several approaches, the first, by modifying the composition either by optimizing the proportions of tungsten and the other matrix ingredients (Ni, Fe, …etc.) or by Adding different types of oxides, like yttrium oxides[6-8], aluminum oxides[9] and zirconium oxides[6]. The second, by optimizing the processing parameters (compaction pressure, sintering temperature, time, and atmosphere). The third approach, by post thermal and mechanical treatments. In this work, the effect of addition of minor amounts of different ceramic oxides on the mechanical and structure properties will be the main concern.

Jing-lian [7], studied the effect of adding dispersed yttrium oxide(Y_2O_3) from 0.02%wt to 0.08%wt to the same tungsten heavy alloy (90W-7Ni-3Fe in %wt), fabricated by the mechanical alloying (MA) process, the powder was then pressed and sintered under H_2 atmosphere. The results showed that a maximum tensile strength of about 1000 MPa and elongation of about 30.8% can be obtained.

Lee et al. [6], investigated the properties of mechanically alloyed oxide dispersion strengthened tungsten heavy alloy 94W-4.8NI-1.2Fe %wt (Ni/Fe weight ratio 4:1), by adding partially stabilized zirconium (PSZ) up to 0.5%wt. The alloy was liquid phase sintered under hydrogen atmosphere. The results confirm that the yield strength and ultimate tensile strength were moderately increased, while elongation was severely decreased.

Demirkan et al. [9], investigated a high tungsten mechanically alloyed composition containing 1%wt Ni and up to 2%wt Al_2O_3 uniaxially pressed and sintered under Ar and H_2 atmospheres. They reported that a remarkable grain refining took place and a sensible increase of ultimate tensile strength and hardness are recorded.

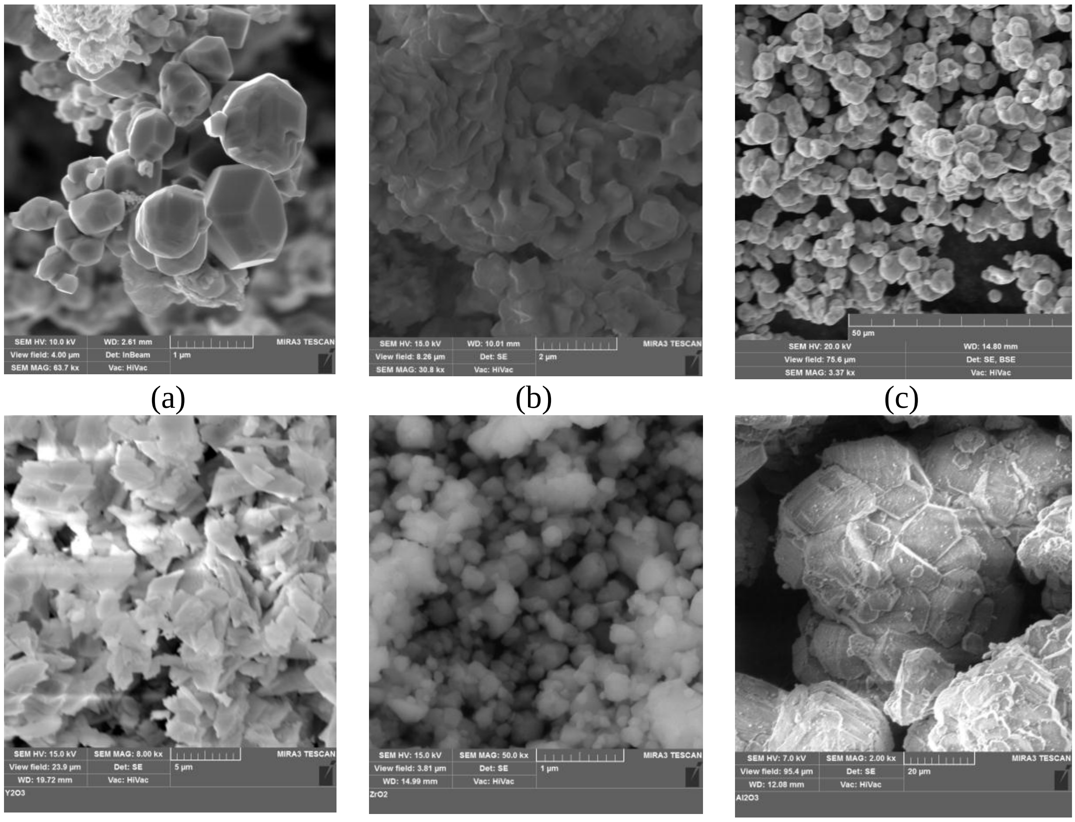
Experimental Work

A heavy tungsten alloy having the composition 90%wt W, 7%wt Ni, and 3%wt Fe was chosen, as a base alloy, to evaluate the effect of limited additions of yttrium oxide (Y_2O_3) from 0.02%wt to 0.5%wt, zirconium oxide (ZrO_2) from 0.1%wt to 0.5%wt, and aluminum oxide (Al_2O_3) from 0.5%wt to 3%wt, on the mechanical and structure properties of this alloy. The characteristics of the elemental and oxide powders are given in Table 1, and the morphologies of the different powders are illustrated in Fig. 1.

The different mixtures with the prescribed basic composition and additives, together with 0.5%wt paraffin wax were mixed in a planetary mixer for 120min.The mixtures were consolidated into green compacts in the form of a standard tensile and impact specimens using uniaxial pressing under 200MPa. The compacted specimens were then liquid phase sintered, in vacuum, at a temperature of 1480ºC for 90min.the regime of the applied sintering cycle is shown in Fig.2 .Density of the obtained sintered specimens was measured by Archimedes water immersion technique. Tensile test was carried out using Instorn tensile test machine type 8032 with loading rate control 8 kN\min. Impact test was carried out using Galdbini charpy pendulum impact testing machine. Hardness values where measured by Rockwell hardness testing machine. The fractures and microstructures of the different specimens were obtained using scanning electron microscope (SEM) type Seo 202.

Table 1: Characteristics of the elemental and the oxide powders

Powder	W	Ni	Fe	Y_2O_3	Al_2O_3	ZrO_2
Shape	polygonal	sponge	Nearly spherical	flake	polygonal	Spherical
Particle size (µm)	1-3	.5-3	1-5	2-3	10-15	0.3-0.4
App density	4.3	3.1	2.6	1.2	0.7- 0.9	2.1
Melting temperature (ºC)	3410	1453	1538	1523	2072	2715
Density	19.3	8.9	7.87	5	4.1	5.68



(a) (b) (c)

(d) (e) (f)

Fig. 1: The morphology of the different used powders. (a) tungsten powder, (b) nickel powder, (c) iron powder, (d) yttrium powder, (e) zirconium powder, and (f) aluminum powder

3. Results and Discussion

3.1 Effect of Yttrium Oxide

3.1.1 Effect on microstructure

The microstructures obtained by scanning electron microscope (SEM), after sintering of the adopted heavy tungsten alloy, with the addition of different percentages of yttrium oxide up to 0.5%wt, are shown in Fig. 3. We can note that the addition of this rare earth oxide has an important effect on refining the tungsten grain size. With increasing the amount of Y_2O_3 the effect of grains pinning increases and the W/W contiguity and connectivity will decrease. Due to the limited added amounts of yttrium oxide, this oxide particles are hardly seen in Fig. 3(a, b, and c) but when it's percentage riches 0.5 %wt they appear as dark small particles at the grain boundaries of tungsten grains, as shown in Fig. 3(d). EDS analyses of these dark particles proved the existence of yttrium.

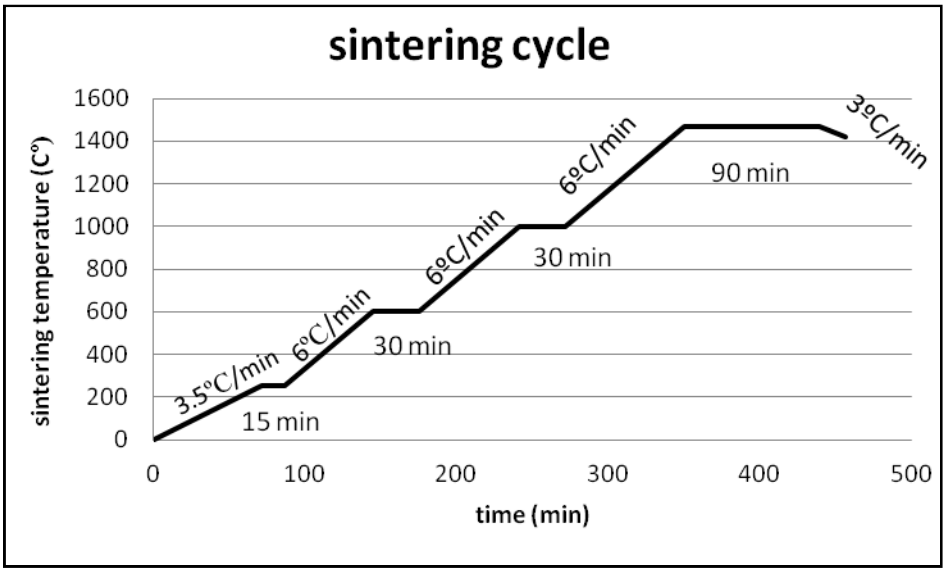


Fig. 2: Sintering cycle for tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

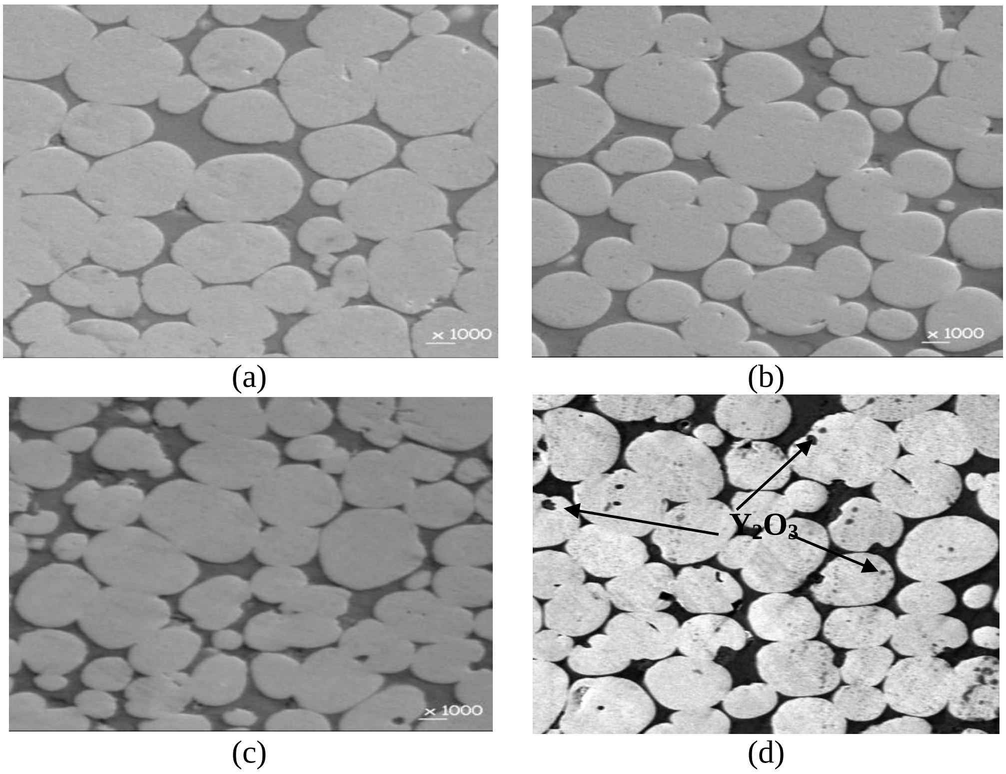
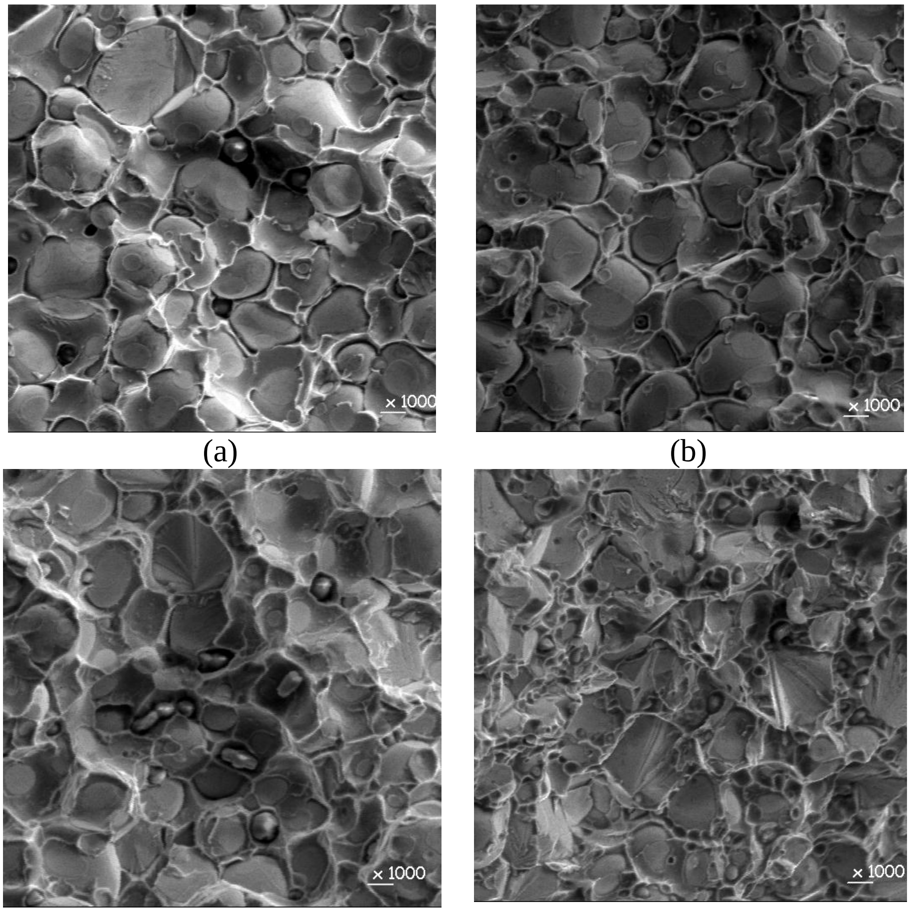


Fig. 3: SEM of tungsten heavy alloy W-7Ni-3Fe, uniaxially compacted under 200 MPa and sintered at 1480ºC for 90min with adding different percentages of Y_2O_3 particles. (a) 0.02%wt, (b) 0.1%wt, (c) 0.3%wt, and (d) 0.5%wt.

3.1.2 Effect on fracture

Due to the complex structure of this liquid phase sintered tungsten heavy alloy, there are several mechanisms of failure, and modes of fracture which can characterize the strength of the bond inside and among the different constituents. There are four fracture paths for the crack to propagate: i) transgranular through tungsten grains by cleavage, ii) matrix failure by ductile dimple mode, iii) tungsten-tungsten intergranular grain boundary separation, and iv) tungsten-matrix interfaces. Typically, all the four features are present simultaneously on the fracture surface. But which one is the dominant that is the main point, for the fracture behavior. The W/W interface is known as the weakest, so fracture generally initiated with that mode type. This type depends on contact area, between tungsten grains, which is known as contiguity phenomena, and is strongly controlled by the tungsten grain size. If the M/W interface is strong, cleavage fracture of tungsten will be the dominant, which generally corresponding to high strength of the alloy. But if M/W interfaces are weak, the W/M separation will be the dominant with less cleavage fracture, which generally corresponding to low ductility and low strength of the alloy.

Fig. 4 illustrates the impact fracture surfaces obtained for the adopted heavy tungsten alloy without and with limited addition of rare earth yttrium oxide (Y_2O_3). We can note that the dominant fracture mode of this alloy, without the addition of yttrium oxide, is the M/W and W/W intergranular separation, as shown in Fig.4a. This indicates lower mechanical properties of the alloy. The addition of only 0.02 %wt yttrium oxide doesn't deeply alter the natural of the fracture modes of the alloy as shown in Fig.4b. Once the percentage of yttrium oxide was increased 0.04 %wt a substantial change of the fracture modes was observed as shown in Fig.4 c. dominant fracture feature. But with adding rare earth oxide (Y_2O_3) with range from 0.02%wt to 0.04%wt the cleavage fracture is the dominant and represent high strength and ductility mechanical properties.



(c) (d)

Fig. 4: Fracture surfaces by SEM of tungsten heavy alloy W-7Ni-3Fe, uniaxially compacted under 200 MPa and sintered at 1480ºC for 90min with adding different percentages of Y_2O_3 particles: (a) 0%wt, (b) 0.02%wt, (c) 0.04%wt, and (d) 0.5%wt.

3.1.3 Effect on physical and mechanical properties

The measurement of the sintered density showed that the addition of the yttrium oxide Y_2O_3, has slightly lowered the relative density of specimens with respect to the theoretical density due to the decreased amount of tungsten in the alloy composition, as shown in Fig. 5.

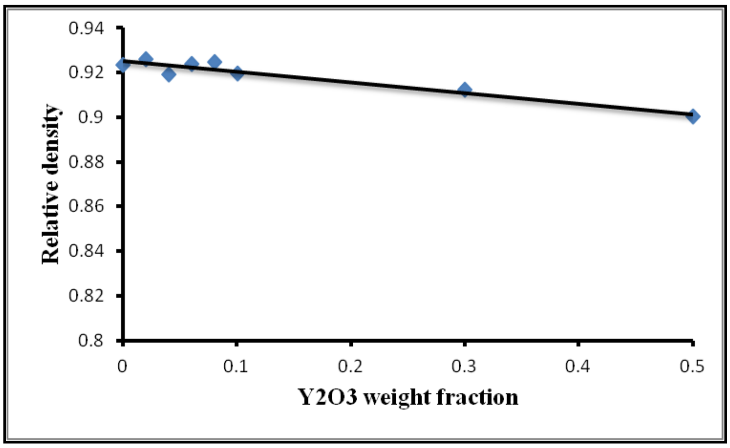


Fig. 5: Effect of limited additions of yttrium oxide on the relative density of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

The obtained mechanical properties, after the addition of yttrium oxide Y_2O_3, to the adopted base alloy, are illustrated in Fig 6. The ultimate tensile strength, ductility, and impact resistance were increased, in a first stage with increasing the percentage of yttrium oxide Y_2O_3 up to 0.04%wt, and then, decreased, in a second stage with further increase in the amount of the oxide of this rare earth metal, as shown in Fig 6 a, b, and c. The peak values of these properties indicated important improvements of about 10%, 25%, and 40% respectively. On the other hand, hardness was continuously moderately increased with increasing the percentage of yttrium oxide Y_2O_3, as demonstrated in Fig 6 d.

These evolutions of mechanical properties of the heavy tungsten alloy 90%wt W, 7%wt Ni, and 3%wt Fe, with the addition of limited amounts of yttrium oxide Y_2O_3, can be explained by the different effects arised by the addition of this oxide. The first, is its effect on refining the grain size and pining of the grain bounders of tungsten grains, which has a deeply favorable effect on improving strength, ductility, and toughness of the alloy. The second, is its effect on strengthening the matrix alloy and then, leading to series embrittlement effect, at relatively higher percentages of this oxide, which results in weakening the interface between matrix and tungsten grain, and consequently decreasing these properties. Moreover, it was reported that [7], when adding the rare earth oxide Y_2O_3 with amount up to (0.02 – 0.04%wt) it works also, in absorbing impurities and oxygen which decreases the structure embrittlement. Furthermore, it was also shown that hardness, as a surface property, is less influenced by the addition of a limited amount of this oxide.

3.2 Effect of Zirconium Oxide

3.2.1 Effect on microstructure

The evolution of the microstructure of the adopted tungsten heavy alloy, with the addition of different amounts of zirconium oxide was studied by SEM, and illustrated in Fig 7. It can be noted that, with increasing ZrO_2 percent up to 0.5%wt, important refining effect can be obtained. On the other hand, contiguity and connectivity are decreased, which allow better wetting of tungsten grains by the matrix liquid phase.

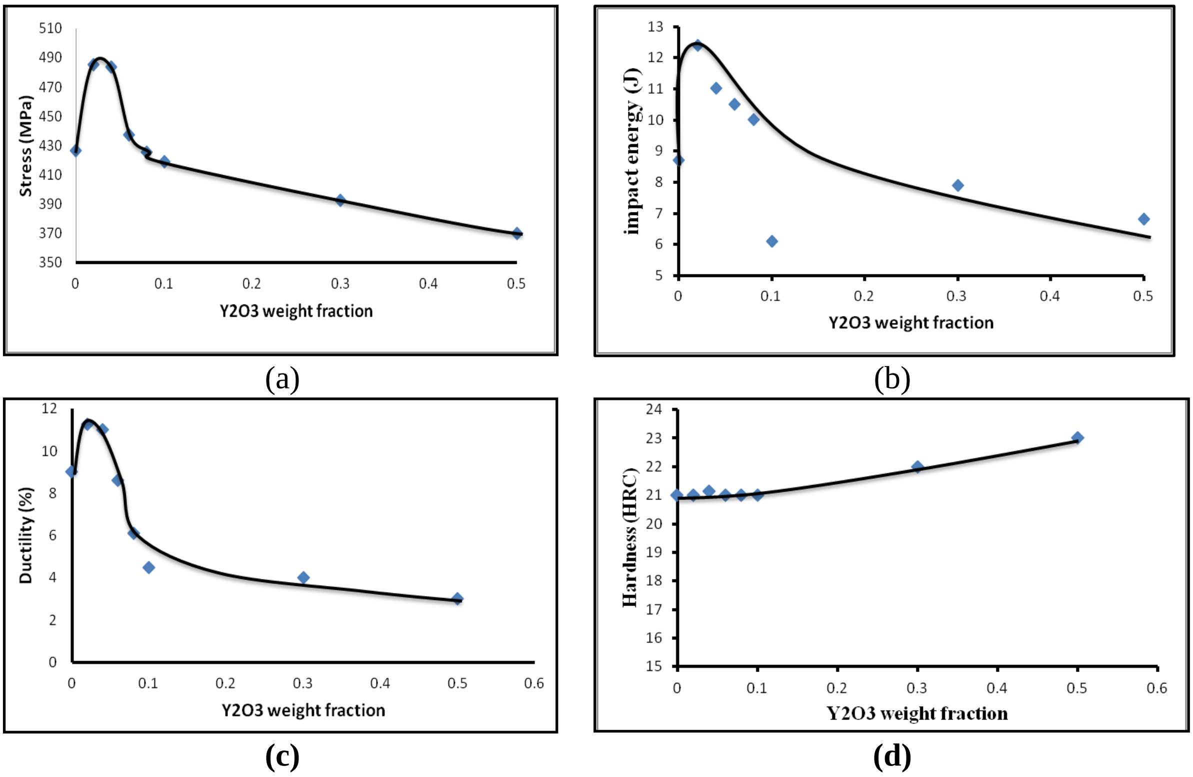
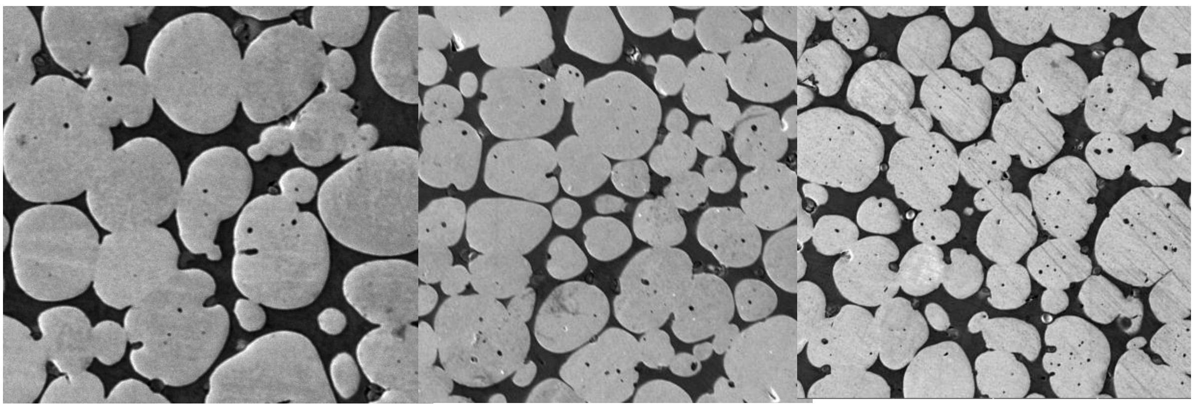


Fig. 6: Effect of limited additions of yttrium oxide on the mechanical properties of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.



(a) (b) (c)

Fig. 7: SEM of tungsten heavy alloy W-7Ni-3Fe, uniaxially compacted under 200 MPa and sintered at 1480°C for 90min with adding different percentages of ZrO_2: (a) 0.1%wt, (b) 0.3%wt, and (c) 0.5%wt.

Figure 8 shows the zirconuim oxide particles on the boundres of tungsten grains and the EDS analysis of these particles which confirms that these black particles refer to the added zirconium oxide.

3.2.2 Effect on fracture

The impact fracture surfaces, obtained for the adopted heavy tungsten alloy, without and with limited addition of zirconium oxide (ZrO_2), are demonstrated in Fig. 9. This alloy, without the addition of zirconium oxide, exhibits dominant coarse intergranular separations through W/W or M/W interfaces, with limited transgranular cleavage, as shown in Fig.9 (a). The addition of increasing percentages of zirconium oxide ZrO_2 does not significantly alter these fracture features, but only led to finer fracture due to its effect on limiting the grain growth, as shown in Fig.9(a, b, and c).

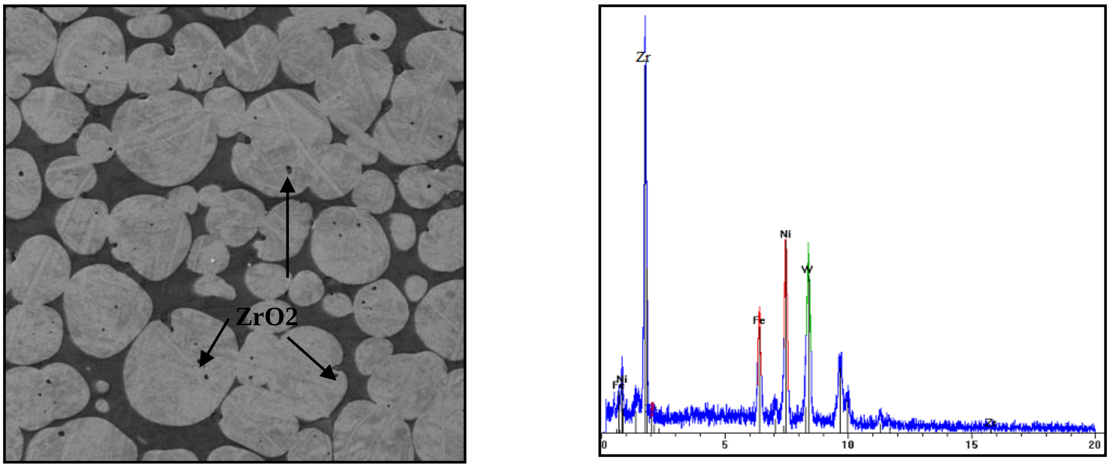


Fig.8: SEM of sintered tungsten heavy alloy with 0.4%wt ZrO2 and the corresponding EDS analysis of the black particles.

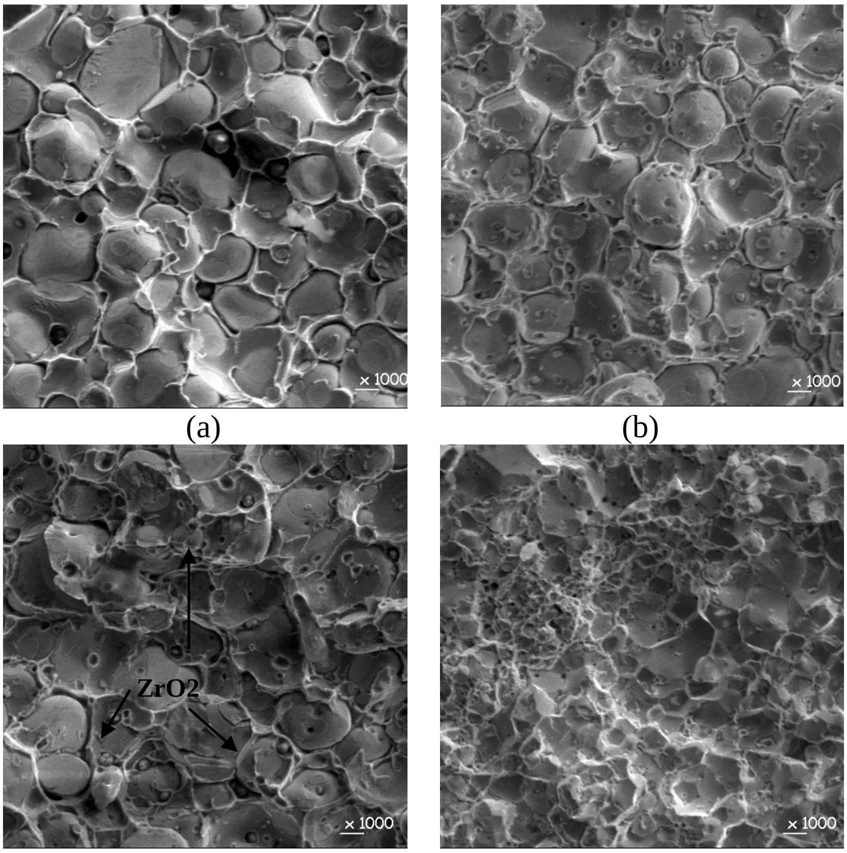


Fig. 9: Fracture surfaces by SEM of tungsten heavy alloy W-7Ni-3Fe, uniaxially compacted under 200 MPa and sintered at 1480°C for 90min with adding different percentages of ZrO_2: (a) 0%wt, (b) 0.1%wt,(c) 0.3%wt, and (d) 0.5%wt.

3.2.3 Effect on physical and mechanical properties

Figure 10 shows the effect of limited additions of zirconium oxide (ZrO_2) on the relative density of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe. The measurement of this density showed an increase of about 1%, with adding zirconium oxide (ZrO_2) up to 0.2%wt. This can be explained by the improved pore closer, due to the addition of this extra fine particle size (0.3-0.4 µm) of this ceramic oxide powder. Further additions of this oxide slightly lower the relative density of specimens. This effect is due to the low density of these oxide particles (5.68 g/c.c), relative to the tungsten particles.

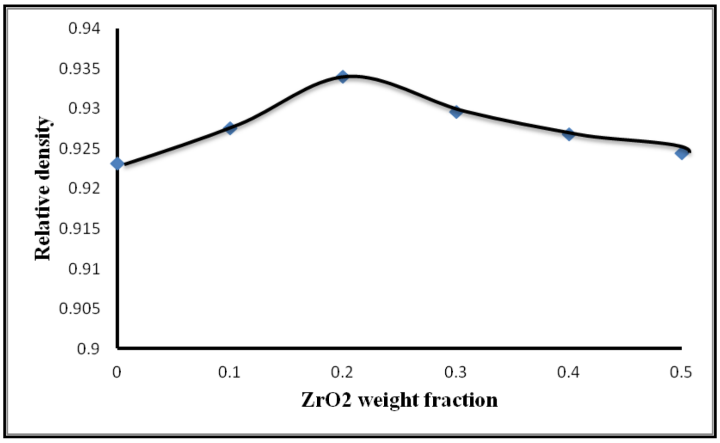
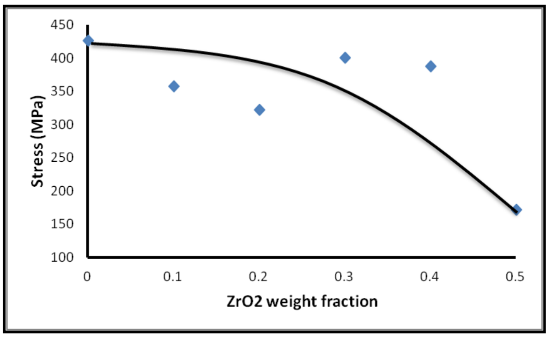
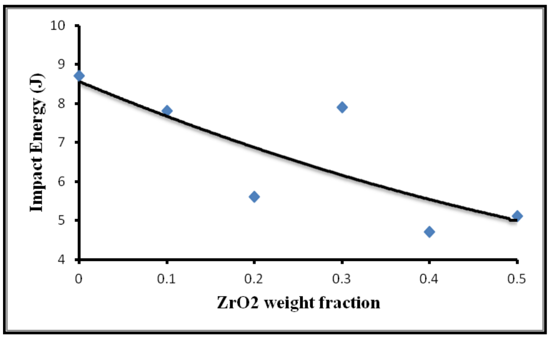


Fig.10: Effect of limited additions of zirconium oxide on the relative density of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

Figure 11 illustrates the obtained mechanical properties, after the addition of zirconium oxide $ZrO_{2,}$ to the adopted base heavy tungsten alloy. The ultimate tensile strength, ductility, and impact resistance were found to decrease, monotonically, with increasing the content of zirconium oxide ZrO_2. On the other hand, hardness increases slightly with the increase of this content.



(a)



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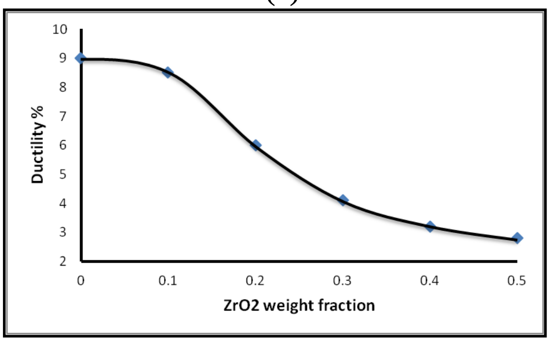
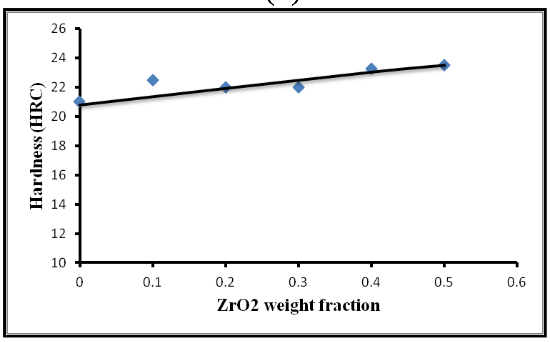


(c)



(d)

Fig. 11: Effect of limited additions of zirconium oxide on the relative density of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

In spite of the strong refining effect of zirconium oxide ZrO_2, its addition, in the pure form, has showed no favorably effect on the mechanical properties of the tungsten heavy alloys, on the contrary, it showed a negative effect. In fact, Zirconia (ZrO_2) was chosen as an excellent engineering ceramic, having important desirable physical, and mechanical properties such as extremely high melting temperature (2680ºC), high strength and fracture toughness. However, the use of this oxide in the pure form, especially, as additive during sintering of heavy tungsten sintered alloys, has no beneficial effect, since it undergoes, during sintering, phase changes very deleterious to the mechanical properties of these alloys, which can explain the drop of the mechanical properties with the addition of this oxide.

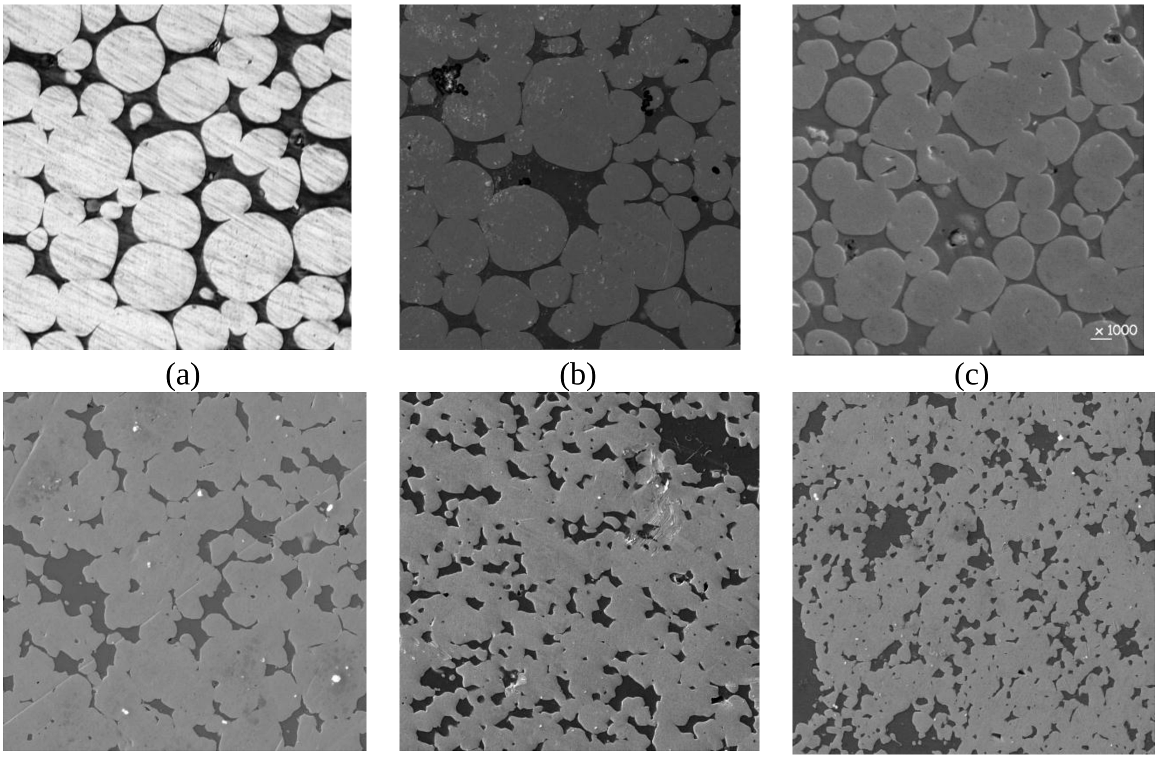
It was reported that [10, 11], at room temperature, zirconia exists on the monoclinic phase. When heated to about 1170°C, it undergoes a phase transformation from monoclinic to tetragonal. This transformation is accompanied with relatively large volume shrinkage of about 9%. Further heating produces another change to cubic at 2370°C. The cubic phase is maintained until the melting point or zirconia is reached 2680°C. On cooling from sintering temperatures and/or high temperature exposure, zirconia undergoes the tetragonal to monoclinic transformation at 950°C and an expansion similar in magnitude of the shrinkage during heat up. This volumetric change associated with that phase transformation is large enough to affect the structural integrity of the material.

It was shown that [12], Stabilization of the tetragonal polymorph of zirconia over wider range of temperatures is accomplished by substitution of some of the Zr^{4+} ions (ionic radius of 0.82 Å) in the crystal lattice with slightly larger ions, e.g., those of Y^{3+} (ionic radius of 0.96 Å). These resulting doped zirconia materials are termed stabilized zirconia. The additions of cubic oxides such as MgO, CaO, Y_2O_3, CeO_2 and other rare earth oxides stabilize the high temperature cubic phase all the way back to room temperature. They also tend to decrease the transformation temperature[13]. In partially stabilized zirconia, similar additions are made, except, not enough to stabilize all of the material, hence the name "partially stabilized zirconia" or "PSZ". These materials typically consist of two or more of the phases cubic, tetragonal and monoclinic. It was also reported that the high temperature yield strength increasing with increasing the content of PSZ dispersoids which act as strengthening agent at high temperature[6].

3.3. Effect of Aluminum Oxide

3.3.1 Effect on microstructure

The effect of adding, different percentages of Al_2O_3, up to 3%wt, to the main tungsten heavy alloy, as reinforcements to the matrix, is illustrated in Fig.12. The structure of the base alloy, without any additions of Al_2O_3, is shown in Fig. 12(a). The comparison of this microstructure with the microstructures obtained, for the same alloy with increasing percentages of Al_2O_3 , Fig. 12(b, c, d, e, and f), indicates significant refining of the grain size. Furthermore, it is clearly observed a strong necking, bridging and welding of tungsten particles as shown in Fig. 12(d, e, and f). The obtained pronounced refining effect, can also be attributed to the strong effect of this oxide on pining of the boundaries of the tungsten particles, which effectively hinder and limit their growth. Demirkan et al. [9] had reported that, Al_2O_3 addition seriously reduce the growth of tungsten grains, specially, when sintering at elevated temperatures. It was also reported that[14, 15], the growth of the tungsten grains, during sintering, takes place by diffusion through two distinct paths. The first, through the liquid phase, by dissolution of tungsten fine grains, and then, precipitation on the coarser grains. The second path, through the existing contact between the tungsten particles, by both, solid state diffusion and simultaneous evaporation and condensation. The observed evolution of the microstructure, demonstrated in Fig. 12(d, e, and f), by the addition of 2, 2.5, and 3%wt Al_2O_3 respectively, may be explained by, the strong effect of Al_2O_3 on suppressing the coefficient of diffusion of tungsten, in the nickel liquid matrix, which retard the growth mechanism by dissolution and precipitation, and allow the solid state diffusion to be the dominant mechanism. Consequently, bridging and welding of tungsten particles become clearly pronounced.



(a) (b) (c)

(d) (e) (f)

Fig. 12: SEM of tungsten heavy alloy W-7Ni-3Fe, uniaxially compacted under 200 MPa and sintered at 1480°C for 90min with adding different percentages of Al_2O_3: (a) 0%wt, (b) 1%wt, (c) 1.5%wt, (d) 2%wt, (e) 2.5%wt, and (f) 3%wt.

Figure 13 shows the aluminum oxide particles on the boundaries of tungsten grains. The white spots indicate the Al_2O_3 particles, which are still on their sites, while the dark spots refer to the removed alumina particles during sample preparation for metallographic observation. The EDS analysis of these particles confirms that these white particles refer to the added aluminum oxide.

3.3.2Effect on physical and mechanical properties

The measurement of the sintered density showed that the addition of the aluminum oxide Al_2O_3, has lowered the relative density of specimens with respect to the theoretical density due to the decreased amount of tungsten in the alloy composition, as shown in Fig. 14. The density is reduced by about 13% when 3% wt of Al_2O_3 was added to the heavy tungsten alloy.

The obtained mechanical properties, after the addition of aluminum oxide Al_2O_3, to the adopted base alloy, are illustrated in Fig 15. The ultimate tensile strength was increased while the ductility and the impact resistance were decreased, in a first stage with increasing of the percentage of aluminum oxide Al_2O_3 up to 1%wt, and then, with further increase in the amount of this oxide, in a second stage, ultimate tensile strength was decreased while ductility and toughness were increased, as shown in Fig 15 a, b, and c. On the other hand, hardness was continuously decreased with increasing the percentage of aluminum oxide Al_2O_3, as demonstrated in Fig. 15d.

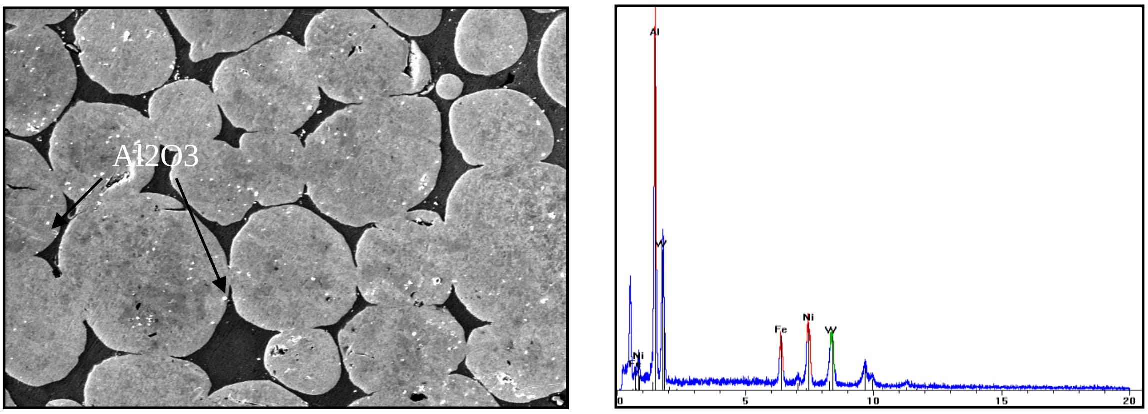


Fig. 13: SEM of tungsten heavy alloy with weight fraction of 1.5%wt Al2O3, and the corresponding EDS analysis of the white particles.

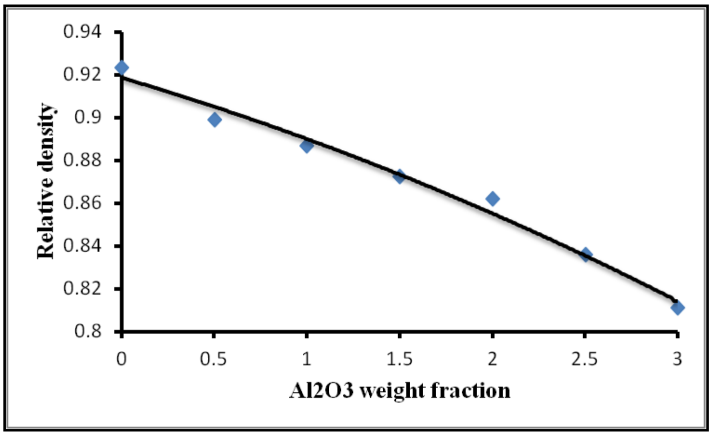


Fig. 14: Effect of different additions of aluminum oxide on the relative density of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

These evolutions of mechanical properties of the heavy tungsten alloy 90%wt W, 7%wt Ni, and 3%wt Fe, with the addition of different amounts of aluminum oxide Al_2O_3, can be explained by the different effects arised by the addition of this oxide. The first, is its effect on refining the grain size and pining of the grain bounders of tungsten grains, which has a favorable effect on improving strength while lowering the values of both ductility, and toughness of the alloy. The second, is its effect on limiting the diffusion of tungsten in the matrix, consequently, its strengthening, which has a considerable effect on improving ductility and toughness of this alloy. On the other hand, this last effect has also lowered the measured hardness on the surface of this alloy.

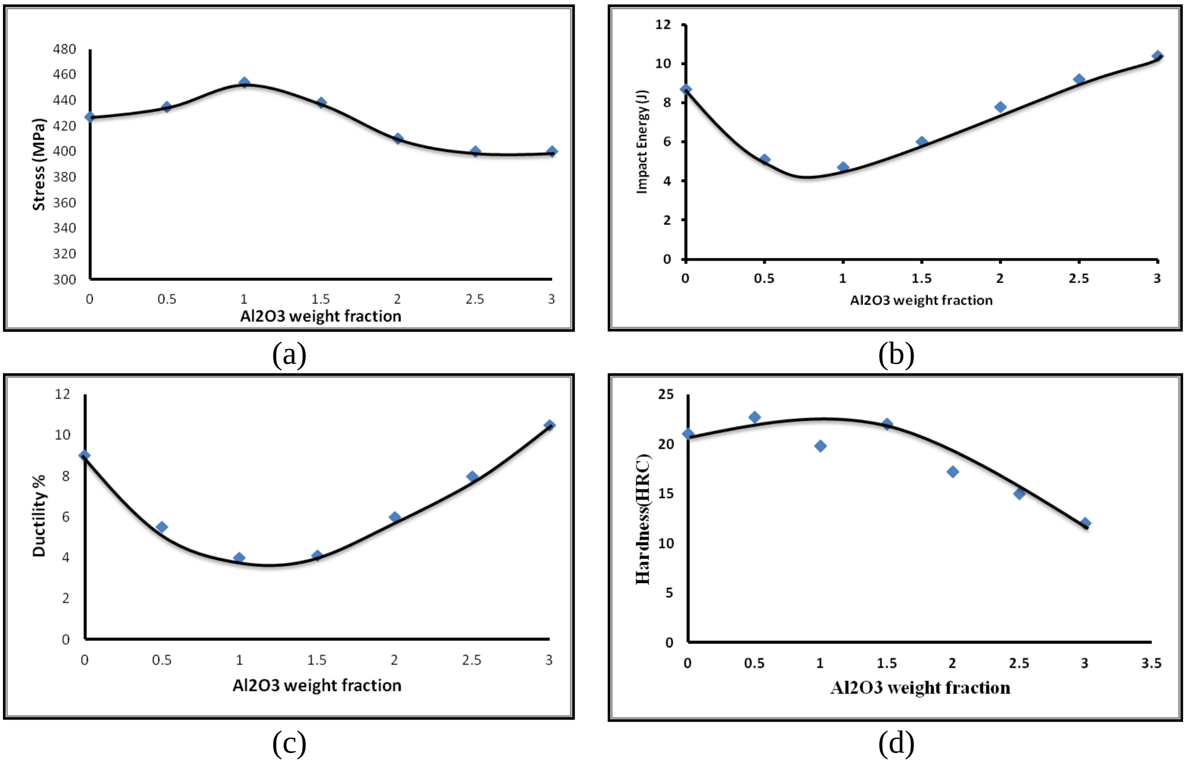


Fig. 15: Effect of different additions of aluminum oxide on the mechanical properties of tungsten heavy alloy 90%wt W, 7%wt Ni, and 3%wt Fe.

Conclusion

1. The addition of limited percentages of yttrium oxide Y_2O_3, up to 0.5%wt, has an important effect on refining the tungsten grain size, by the grains pinning effect during growth, moreover, W/W contiguity and connectivity are decreased.
2. The addition of 0.04%wt of yttrium oxide Y_2O_3, has favorable effect in improving the ultimate tensile strength, ductility, and impact resistance, of the tungsten heavy alloy (90W-7Ni-3Fe). This added percentage, of that rare earth metal oxide, secure about 10%, 25%, and 40% improvements, in these mechanical properties respectively. Further increase of the amount of this oxide lowers these properties.
3. The addition of limited amounts of yttrium oxide Y_2O_3, develops the dominant fracture mode, of tungsten heavy alloys, from the intergranular to cleavages mode.
4. When zirconium oxide ZrO_2 is added, with percentages up to 0.5%wt, to the same tungsten heavy alloy (90W-7Ni-3Fe), a strong refining effect can be obtained, however, it has a negative effect on the mechanical properties.
5. The addition of 1%wt of alumina oxide, to the same tungsten heavy alloy, improves the ultimate tensile strength by about 7%, the ductility and the impact resistances were decreased. On the contrary, higher additions, of this oxide, decrease the tensile strength and enhance the ductility and toughness of this alloy. Moreover, the relative density of the alloy decreases by about 11% when the percentage of this oxide attains 3%wt.

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