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Microstructural evolution on the sintered properties of W-8 pct Mo-7 pct Ni-3 pct Fe alloy

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The microstructure of W-8 pct Mo-7 pct Ni-3 pct Fe alloy has been found to evolve in concomitance with the reprecipitation of tungsten atoms from the matrix phase onto the grains. With increase in isothermal holding time at 1480°C, the concentration of tungsten in the matrix phase reduces from 53.7 wt pct for an isothermal holding time of 5 minutes, and gradually approaches an equilibrium value of about 18 wt pct. The continuous solid-liquid reactions result in gradual penetration of the liquid phase into the boundaries of the skeletal solid grains. Consequently, contiguity of the tungsten based grains drops sharply during the initial stage of isothermal holding, and reaches minimum after an isothermal holding time of 120 minutes. The contiguity subsequently increases again as the coalescence of grains commences. Simultaneous diminishing of the concentration of tungsten in the matrix phase along with decrease in contiguity of the grains result in an optimal combination of mechanical properties after an isothermal holding time of 120 minutes. Molybdenum has been found to bring about a substantial microstructural refinement, whereby a mean grain size of only 22 μm is attained from an isothermal holding time of as long as 240 minutes.

1. Introduction

Mechanical properties of W-Ni-Fe heavy alloys are well known to be modified by the addition of molybdenum, which has a mutual solubility in the solid grains as well as in the liquid matrix phases [1–6]. It is indicated that alloying of W-Ni-Fe alloys with molybdenum results in a lower solubility of tungsten in the liquid matrix phase, yielding refined microstructures characterized by bimodal grain size distribution and jagged grain shape [1–5]. Due to both grain size refinement and solid solution strengthening of the liquid matrix phase by molybdenum [3], the strength and hardness of W-Mo-Ni-Fe alloys steadily increases with increase in the addition of molybdenum up to a concentration of 16 wt pct [1, 5]. Although a trade-off exists between strength and ductility, as in various conventional alloys, the performance of the W-Mo-Ni-Fe heavy alloys approaches that of W-Ni-Fe alloys subjected to moderate levels of swaging and aging. Hence, W-Mo-Ni-Fe alloys possibly possess the capability of net-shape or near-net-shape forming.

With a limited range of processing parameters having been investigated so far, bimodal grain size distribution and jagged grain shape have been deemed as the sole characteristics of W-Mo-Ni-Fe alloys [1–5], while spheroidal grains analogous to those of W-Ni-Fe alloys have also been observed [6]. The reason for this discrepancy lies in the fact that different sintering temperatures were used in these studies, with higher sintering temperatures yielding more spheroidal grains. In fact, spheroidization of the grains has been clearly shown to occur with increase in sintering temperature [4], indicating that the state characterized by jagged grains is just a transient state during microstructural evolution. The existence of jagged grains is due to a slow spheroidization rate of the solid grains arising from the fact that the mass transfer of tungsten atoms across the solid-liquid interfaces is an interfacial-reaction-controlled mechanism [7]. It is thus expected that the microstructural characteristics of W-Mo-Ni-Fe alloys can differ substantially from those of W-Ni-Fe alloys under the same sintering conditions.

Microstructural characteristics such as density, grain size, and interconnection among solid grains are well known to affect the mechanical properties of tungsten based heavy alloys [8]. As Mo retards the microstructural evolution rate of W-Ni-Fe alloys, the mechanical properties of W-Mo-Ni-Fe alloys are expected to be different from those without Mo when they are sintered under the same conditions. The aim of this study was to

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investigate microstructural evolution and its correlation with the sintered properties of W-8 pct Mo-7 pct Ni-3 pct Fe alloy, by varying the isothermal holding time at a sintering temperature of 1480°C. A temperature of 1480°C is a very common sintering temperature for W-Ni-Fe alloys, but is towards the lower end of those usually employed for sintering W-Mo-Ni-Fe alloys, which range between 1475°C and 1560°C [1–6]. The present low sintering temperature was chosen to enable better freezing of the kinetic processes by reducing the repartitioning rates of the elements, and consequently, easier observation of the intermittent developments of microstructure and properties.

2. Experimental

The heavy alloy used in this study was W-8 pct Mo-7 pct Ni-3 pct Fe by weight, prepared by using mixed elemental powders. The characteristics of the constituent elemental powders are presented in Table I. The powders were initially blended and wet-milled in a plastic jar for 16 hours, using 304 stainless steel balls (diameter = 3 mm). The milling medium was composed of heptane and paraffin wax (1.5 wt pct of powder). Subsequent to milling, the slurry was dried, granulated, and sieved with a screen (70 mesh). Standard tensile specimens were prepared by pressing the sieved granules at a pressure of 110 MPa. The specimens were then sintered in a tube furnace, wherein the thermal profile was so adjusted as to perform the functions of binder removal, oxide reduction, and powder sintering. The binder burnout profile comprised heating at a rate of 3 K/min to 350°C, hold for 1 hour, further heating at a rate of 3 K/min to 500°C, and again hold for 1 hour. This schedule was followed directly by heating at a rate of 10 K/min to 1000°C, and holding for 1 hour, to reduce the possible formation of an oxide layer on the powder surface. Finally, the sintering profile comprised heating at a rate of 10 K/min to 1480°C, and holding for either 5, 15, 30, 60, 120, 180, or 240 minutes. Hydrogen atmosphere was maintained until the last 10 minutes (or 5 minutes for an isothermal hold of 5 minutes) of the isothermal hold, when the atmosphere was changed to argon.

The sintered specimens were polished to a 0.3 μm finish without etching for metallographic observation. Determination of mean intercept length and contiguity of grains were carried out over more than 200 grains. The mean grain size was defined as the value of the mean intercept length multiplied by 1.5. Contiguity was measured metallographically by counting the number of intercepts per unit length of a test line, N, and is defined as [8]

$$C = \frac{2N_{\mathrm{W-W}}}{(2N_{\mathrm{W-W}} + N_{\mathrm{W-L}})}$$

where the subscripts W-W and W-L denote the boundaries between tungsten grains and interfaces between tungsten grains and the liquid matrix phase, respectively. The tensile specimens were lapped on two flat faces to a 240 grit finish. The tensile tests were carried out with 20 mm gauge length at a strain rate of 0.004 mm/s. True stress at fracture was considered as the ultimate tensile strength (UTS). The hardness was measured on the Rockwell A scale. All mechanical data reported in this article are the averages of five measurements. Quantitative elemental analysis of the matrix and tungsten based phases was carried out by electron probe microanalysis (EPMA), with an excitation voltage of 20 keV. The recorded intensities of Ni K$_\alpha$, Fe K$_\alpha$, Mo L$_\alpha$, and W L$_\alpha$ radiations were used for calculating the concentrations of nickel, iron, molybdenum, and tungsten, respectively. The uncertainty in this composition analysis was about ±1.5 wt pct and the effective excitation volume was about 1.5 μm in diameter. Therefore, the determination of phase composition was carried out only for domains larger than 2 μm in size, in order to avoid interference from the adjacent domains.

TABLE I Characteristics of the elemental powders used in this study

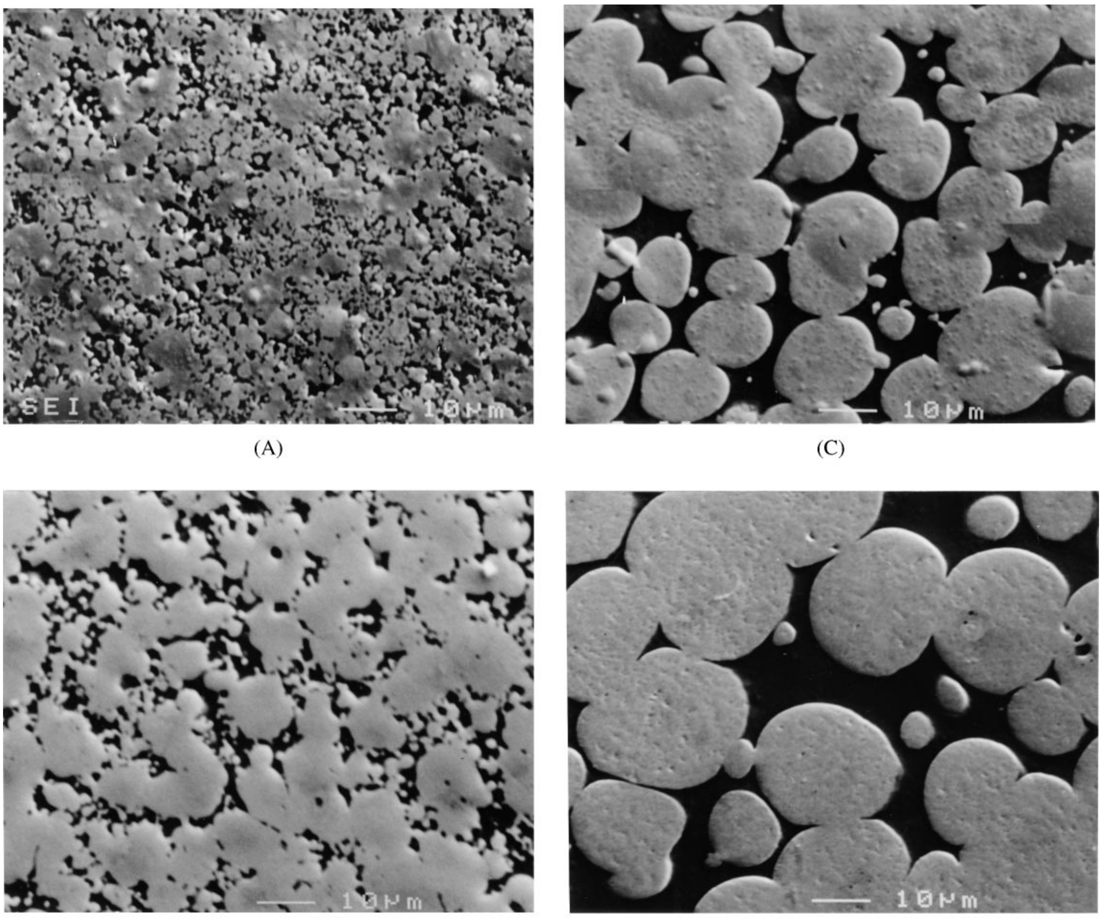
Property	W	Mo	Ni	Fe
Vendor	Korea Tungsten	Climax	INCO	BASF
Designation	KM-8	OMP-6	123	OM
Purity, wt pct	99.95	99.8	99.8	97.8
Mean particle size, μm	4	5	11	4
Tap density, g/cm^3	2.8	2.2	4.7	4.3
Major impurities	O	Si, Fe, Sn	C, O, Fe	N, C, O

3. Results and Discussion

3.1. Microstructural Characteristics

Fig. 1 shows the evolution of microstructure with increase in isothermal holding time. Pores could hardly be found for an isothermal holding time as short as 5 minutes. In fact, nickel and molybdenum form a eutectic liquid phase at 1318°C [9]. This liquid phase assists full densification of the alloy at a sintering temperature as low as 1350°C [7]. It is noted that the evolution of microstructure can be roughly separated into two stages, divided by a holding time of between 30 and 60 minutes. The tungsten based solid phase was composed of fine dispersed grains and irregular skeletal domains in the early stage of isothermal hold, and spheroidal isolated grains in the latter stage of isothermal hold. In comparison, two discrete stages associated with dramatically different microstructural characteristics after the formation of the liquid phase have also been identified for W-8 pct Ni, wherein the early stage ended rather abruptly after 2 to 3 minutes of sintering at 1550°C [10].

Fig. 2 shows the microstructure of a specimen sintered for 30 minutes, along with its dot mapping of molybdenum by EPMA. It can be seen that the large skeletal domains possess higher concentrations of molybdenum. This observation suggests that, subsequent to the formation of the liquid phase, the tungsten grains that are sinter-bonded by nickel or iron decompose into fine dispersed grains surrounded by the liquid phase, whereas those sinter-bonded by molybdenum retain their skeletal structure. Owing to this fact, bimodal



(A) (C)

(B) (D)

Figure 1 Microstructure of the W-8 pct Mo-7 pct Ni-3 pct Fe alloy sintered at 1480°C for (A) 5, (B) 30, (C) 60, and (D) 240 minutes.

distribution of grain size evolves in the early stage of microstructural evolution. In fact, elements that have an infinite solubility in tungsten, such as tantalum, and elements that have a low solubility in tungsten, such as rhenium, are also known to enhance the sinter-bonding of tungsten grains and result in bimodal distribution of grain size [1–3, 11].

The dramatically different microstructural characteristics with the extended isothermal hold indicated that the microstructure comprising fine dispersed grains and irregular skeletal domains does not belong to the state of thermodynamic equilibrium. Such a kinetic process can also be found when the tungsten concentration in the liquid matrix phase is analyzed. Fig. 3 shows that the matrix phase is super-saturated with tungsten atoms in the early stage of microstructural evolution, which gradually leveled off and approached an equilibrium value as the latter stage of microstructural evolution commenced. Differential lattice defect concentration is known to result in supersaturation of the bulk solid phase in the liquid phase, which yields precipitation with fewer defects [12]. For the alloy investigated in this study, the reprecipitation of tungsten atoms from the liquid matrix phase toward the tungsten based grains proceeded following a first order interfacial reaction controlled process [7]. Declining concentration of tungsten in the matrix phase brings about a gradual change in the interfacial properties between the grains and the matrix phase during the solid-liquid reaction,

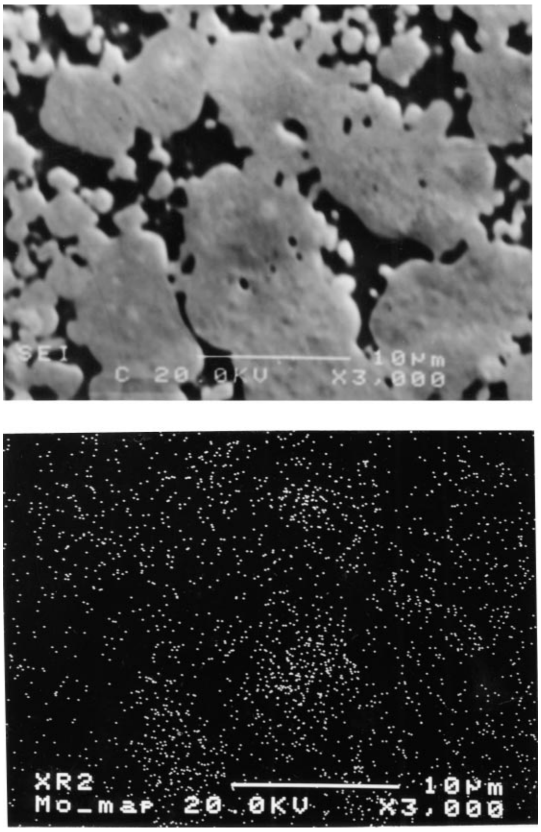


Figure 2 Enlarged microstructure of the W-8 pct Mo-7 pct Ni-3 pct Fe alloy sintered at 1480°C for 30 minutes and the corresponding molybdenum dot mapping by EPMA.

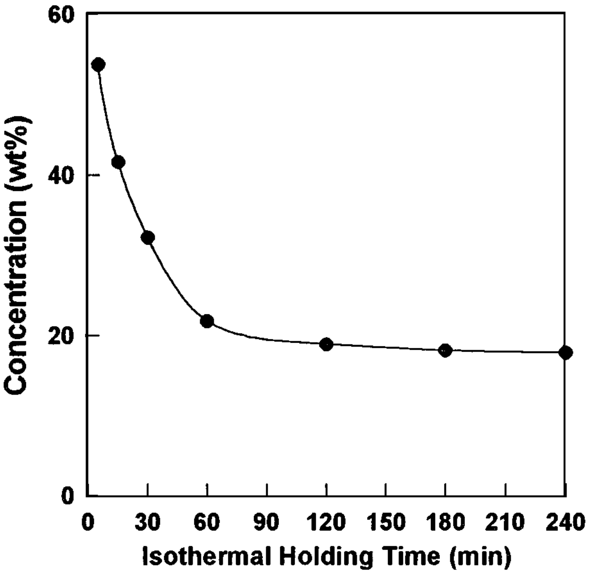


Figure 3 Variation in the concentration of tungsten in the matrix phase as a function of isothermal holding time.

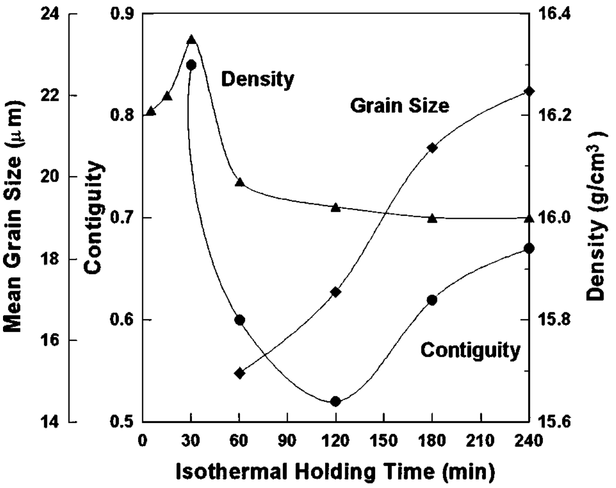


Figure 4 Variations in the sintered density, mean grain size, and contiguity of grains as a function of isothermal holding time.

and also in the viscosity of the liquid phase. These, in turn, cause progressive penetration of the liquid phase into the grain boundaries of the skeletal solid phase and result in the disintegration and subsequent gradual spheroidization of these grains with extended isothermal hold. Hence, it appears quite clear that the existence of jagged grains is only a transient state during the evolution of microstructure from a highly interconnected skeletal structure into fully spheroidal grains. In fact, spheroidal grains have been found to dominate the microstructure of W-Mo-Ni-Fe alloys sintered at higher temperatures, such as 1530°C for 30 minutes [5], and 1540°C for 30 minutes [6].

Fig. 4 shows the variations in sintered density, mean grain size, and contiguity of tungsten grains as a function of isothermal holding time. The sintered densities of the specimens held at the sintering temperature for 5, 15, and 30 minutes are slightly higher than the theoretical density of 16.11 g/cm^3 predicted by the ideal law of mixtures, whereas those for the specimens held at the sintering temperature for 60, 120, 180, and 240 minutes are in the range of 99.3 pct to 99.8 pct of 16.11 g/cm^3. In addition to the possible uncertainty in measurement, a sintered density being higher than that predicted by the ideal rule of mixtures was owing to two facts found in a previous study [7]. First, the matrix phase that was super-saturated with tungsten atoms in the early stage of isothermal hold had a lattice parameter negatively deviating from the ideal law of mixtures (Vegard's law). This resulted in a real density of the matrix phase being higher than the density predicted by the rule of mixtures. Second, the volume fraction of liquid matrix phase in the duplex microstructure dropped from 0.31 to 0.23 as the isothermal holding time increased from 5 minutes to 60 minutes. A denser matrix phase with a larger volume fraction yielded a density higher than that predicted by the rule of mixtures.

The mean grain size of the tungsten based grains steadily increases during the latter stage of microstructural evolution, but with a growth rate relatively lower than that for W-Ni-Fe alloys. For example, the mean grain size of W-8 pct Mo-7 pct Ni-3 pct Fe alloy sintered at 1480°C for 240 minutes was 22 μm, while that of W-4.9 pct Ni-2.1 pct Fe alloy sintered at 1480°C for 40 minutes was also 22 μm [13]. Such a comparison is valid due to the fact that these two alloys had approximately equivalent volume fractions of the liquid matrix phase, and were synthesized using the same tungsten, nickel, and iron powders. Thus, molybdenum indeed impedes the growth of tungsten based grains. Previous report indicated that the rate of grain growth of the same alloy follows an approximate square root time dependence in the initial stage of isothermal hold [14]. The grain growth kinetic is thus an interfacial reaction controlled process [7, 14]. The impediment of grain growth with the existence of molybdenum is the consequence of the counter-transport of tungsten and molybdenum atoms across the interface between solid and liquid phases [7]. In this interfacial reaction controlled process, molybdenum atoms in the tungsten based grains dissolve into the liquid matrix phase while tungsten atoms reprecipitate from the liquid matrix phase toward the tungsten based grains.

On the other hand, the contiguity of the grains also behaved differently. With the penetration of the liquid phase into the grain boundaries, the grains of skeletal structure gradually separate and alter their shapes during the solid-liquid reaction, resulting in a substantial decrease in the contiguity of the solid phase. The contiguity of grains reaches a minimum when spheroidization of the grains fully develops. With further isothermal hold, coalescence of the grains becomes so severe that contiguity of the grains slightly increases. A similar trend has also been reported in the literature for W-8 pct Ni alloy, only with contiguity of the grains leveling off for longer holding times [10]. The decrease of contiguity in the early stage of isothermal hold is ascribed to the continuous change in interfacial properties with increase in isothermal holding time [8, 10]. In the present study, such continuous change in the interfacial properties with increasing isothermal holding times is closely associated with the decrease of tungsten concentration in the liquid phase, as indicated in Fig. 3.

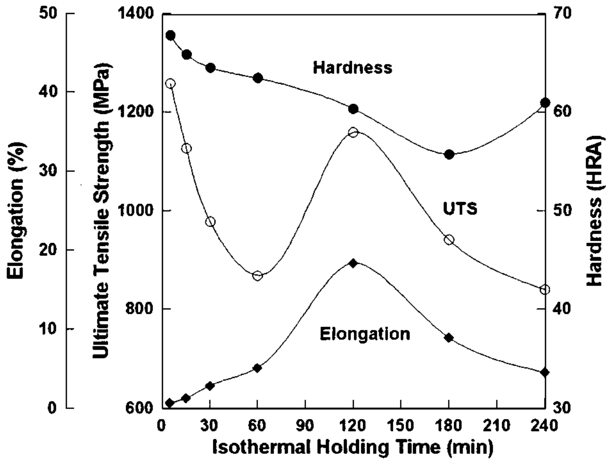


Figure 5 Variations in the hardness, ultimate tensile strength, and tensile elongation as a function of isothermal holding time.

TABLE II A comparison of the processing conditions and mechanical properties of W-8 pct Mo-7 pct Ni-3 pct Fe alloy

Sintering Temp. (°C)	Holding Time (min)	Cooling Method	UTS (MPa)	Elong. (%)	Hardness (HRA)	Ref.
1480	120	furnace cooling	1195	21	60	this study
1500	30	water quench	1030	20	66	1, 3, 5
1475	30	furnace cooling	1144	8	68	4*
1500	30	furnace cooling	1115	20	64	4*
1530	30	furnace cooling	1067	27	64	4*

*W-8 pct Mo-8 pct Ni-2 pct Fe, powder injection molding.

3.2. Mechanical properties

Fig. 5 shows the variation in mechanical properties as a function of isothermal holding time. The hardness initially drops from a value of 68 HRA to 56 HRA and then slightly rises to 61 HRA. The supersaturation of tungsten atoms in the matrix phase, as shown in Fig. 3, is responsible for the high hardness for specimens experiencing a short isothermal hold. With a longer isothermal hold, the concentration of tungsten in the matrix phase diminishes, which reduces the solution hardening effect of tungsten on the matrix phase and hardness reaches a minimum [6]. A similar observation regarding the variation of hardness of tungsten heavy alloy with the alloying of molybdenum and increase of isothermal holding time can be found elsewhere [15]. In this cited report, enhanced hardness in the initial stage of isothermal hold is ascribed to the solution hardening effect of molybdenum on both the grains and matrix, and the decrease of hardness with increase in isothermal hold is attributed to grain growth. With further isothermal hold, tungsten based grains gradually develop into a structure with high contiguities, which resists the plastic deformation of the composite structure more efficiently and the hardness rises slightly [16].

The variation in tensile elongation shows a trend contrary to that of contiguity. In the early stage of isothermal hold, the tungsten atoms in the matrix phase gradually reprecipitate toward tungsten based grains and the matrix phase progressively penetrates into the boundaries of skeletal tungsten grains. Consequently, the ductility of the alloys increases and reaches a maximum value of 21% for an isothermal holding time of 120 minutes. With a longer isothermal hold, the concentration of tungsten atoms in the matrix phase has leveled off and the increase in contiguity of tungsten based grains retards plastic deformation prior to fracture and the ductility decreases. On the other hand, the ultimate tensile strength exhibits a slightly different trend. The alloy initially behaves similar to pure tungsten material, exhibiting high strength, yet low ductility. With the penetration of the liquid phase into the grain boundaries of tungsten based grains, the tensile strength drops slightly but the ductility increases substantially. The decrease of strength in the early stage of isothermal hold is due to several possible interacting factors, such as increase in grain size, loss of skeletal structure of the tungsten based grains, and declining concentration of tungsten in the matrix phase. With further increase in isothermal holding time, the strength of the alloy shows a trend analogous to that of ductility. The ultimate tensile strength of the alloy reaches a peak value of 1195 MPa for an isothermal holding time of 120 minutes.

The optimal combination of mechanical properties achieved in this study is compared in Table II with those reported in the literature [1, 3–5]. It can be seen from Table II that, with the same isothermal holding time, increasing the sintering temperature reduces the hardness and enhances the ductility of W-8 pct Mo-8 pct Ni-2 pct Fe alloy. This behavior is analogous to that due to increase in isothermal holding time up to 120 minutes, found in this study. It is also noted that a high cooling rate subsequent to sintering, such as water quenching, enhances the hardness, ultimate tensile strength, tensile elongation, and toughness of heavy alloys [17, 18]. Although without water quenching, the optimal combination of strength and ductility achieved here for an isothermal holding time of 120 minutes is encouraging, as it is towards the high end of the range of combined strength and ductility. The hardness value belonging to the optimal combination of strength and ductility in this study (60 HRA) is lower than the values reported in past investigations. Only for shorter isothermal holding times are the hardness values achieved in this study comparable to the values reported in past investigations. It is generally believed that under the same sintering conditions, alloying with molybdenum increases the hardness of heavy alloys [1–5], a result believed to be ascribed to the solution hardening effect of molybdenum on both matrix as well as grain phases. In comparison, the hardness values of W-4.9 pct Ni-2.1 pct Fe alloy processed under various conditions range from 61 to 65 HRA [19]. This alloy has a volume fraction of matrix phase approximately equivalent to that of the alloy investigated in this study. It is thus clear that increase in hardness with increased molybdenum alloying is not a necessary result. It is also caused by the reduced volume fraction of the matrix phase, when tungsten is partially substituted by molybdenum at equivalent weight fractions instead of equivalent volume fractions.

In the present study, the optimal combination of strength and ductility was achieved when the microstructure was composed of spheroidal grains, instead of jagged grains that are commonly observed [1–5]. It is thus clear that there exists a window of processing parameters, that corresponds to a microstructure comprising grains with low contiguities and a matrix phase with low concentrations of tungsten. Within this window, the optimal combination of strength and ductility can be attained. The mechanical properties, associated with the microstructure comprising jagged grains, may not correspond to the optimal combination. Indeed, for W-Mo-Ni-Fe alloys, a higher sintering temperature or a longer isothermal holding time is required to develop spheroidal microstructures equivalent to those of W-Ni-Fe, as the kinetic process is hindered by the incorporation of molybdenum. For example, for W-12 pct Mo-7 pct Ni-3 pct Fe alloy sintered at 1500°C, extending the isothermal holding time from 30 to 120 minutes results in a slight decrease in tensile strength from 1100 to 1025 MPa, but dramatically enhances the ductility from 10 to 19 pct [1]. This is due to enhanced spheroidization and reduced contiguity of the grains. Similar trends have also been reported for W-Mo-Ni-Fe alloys with molybdenum content of up to 16 wt pct, when the sintering temperature was raised from 1475°C to 1530°C [4], or when the isothermal holding time at 1500°C was extended from 30 to 120 minutes [5].

Fig. 6 shows the fracture surfaces of specimens held at the sintering temperature for 30 and 120 minutes. These specimens indeed exhibit different grain sizes and fracture modes. The lack of ductility for an isothermal holding time of 30 minutes is obvious. Although the tungsten and molybdenum grains are sinter-bonded to close to full density, grain growth appears to be not very active in the early stage of microstructural evolution, as fine grains with sizes in the neighborhood of 2 μm predominate on the fracture surface. In addition, the matrix phase can hardly be differentiated from the tungsten based grains, reflecting the lack of ductility of the matrix phase and incomplete penetration of the liquid phase into the grain boundaries. This lack of ductility of the matrix phase owes to the supersaturation of tungsten in the matrix phase in the early stage of microstructural evolution. The grains of the specimen held at the sintering temperature for 120 minutes are significantly larger than a specimen held for 30 minutes. The fracture surface of this specimen can be seen to be composed of various fracture modes, including cleavage of grains, fracture along the interface between grains and matrix phase, ductile fracture of matrix phase, and fracture along the grain-grain interface. It has been pointed out previously that high strength and ductility are associated with the enhanced cohesion strength between the grains and the matrix phase, characterized by a fracture surface comprising cleavage of grains and ductile matrix phase fractures [15, 20]. Failure along the weakest grain-grain interface can be seen to occupy a large portion of the fracture surface, a result of high contiguity of the grains (larger than 0.5) that inevitably induces fracture along the grain-grain interface. Hence, further optimization of the processing parameters to reduce the contiguity of grains appears to be a viable route to further enhance the performance of W-8 pct Mo-7 pct Ni-3 pct Fe alloy.

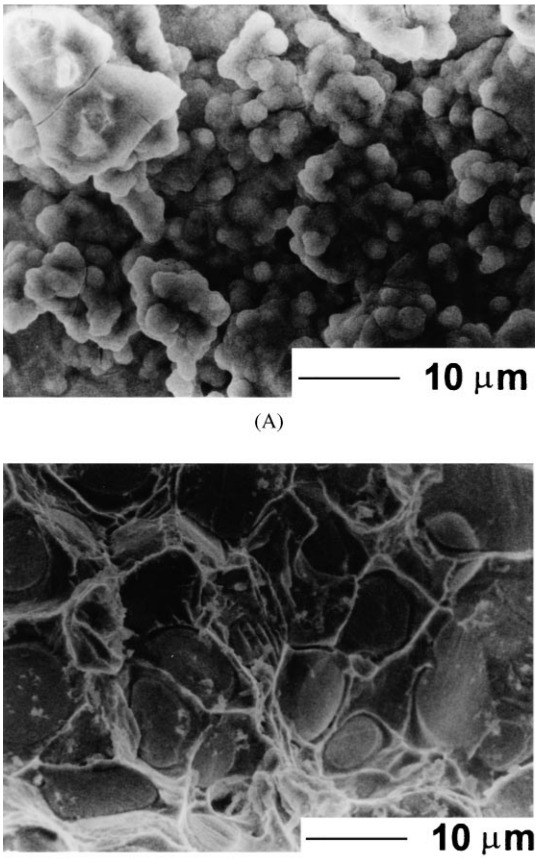


Figure 6 Fracture surfaces of W-8 pct Mo-7 pct Ni-3 pct Fe alloy sintered at 1480°C for (A) 30, and (B) 120 minutes.

4. Conclusion

Microstructural evolution of W-8 pct Mo-7 pct Ni-3 pct Fe alloy was investigated by varying the isothermal holding time at a sintering temperature of 1480°C. Two distinct stages of microstructural evolution could be identified subsequent to the formation of the liquid phase. In the initial stage (5 to 30 minutes isothermal hold), the liquid matrix phase was super-saturated with tungsten atoms, and the tungsten based solid phase was composed of fine dispersed grains and irregular skeletal domains. Concomitant with the reprecipitation of the super-saturated tungsten atoms from the matrix phase toward the solid grains in the later stage, the liquid phase also gradually penetrated into the grain boundaries within these skeletal domains, bringing about spheroidization of grains. Mechanical properties of the resulting alloy were found to strongly depend on the microstructural details and behave in a complicated manner. The alloy behaved as a hard, strong, and brittle material in the initial stage of isothermal hold, due to the supersaturation of tungsten atoms in the liquid matrix phase and a high degree of contiguity (interconnection among solid grains). The alloy gradually developed into a strong but ductile material as the concentration of tungsten in the liquid matrix phase decreased and the solid grains became isolated and spheroidized. An optimal combination of strength and ductility was achieved when the grains fully developed into spheroidal shapes with lowest contiguity, in contrast to the state with jagged grains reported in previous studies.

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