THE FEASIBILITY STUDY OF W-CU COMPOSITE PRODUCTION BY SUBMICRON PARTICLES ADDITION AND INFILTRATION

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Abstract: *In this paper the feasibility of fabricating controlled porous skeleton of pure tungsten at low temperature by addition of submicron particles to tungsten powder (surface activated sintering) has been studied and the best parameters for subsequent infiltration of Cu were acquired. The effects of addition of submicron particles and sintering temperature on porous as well as infiltrated samples were studied. The samples were examined by scanning electron microscopy (SEM), Vickers hardness measurements and tensile test. The composites made have been investigated and revealed the making W-Cu composite with good density, penetrability, hardness and microstructure. Consequently, the sintering temperature was reduced considerably (Ts≤1650oC) and a homogeneous porous tungsten was obtained. Also, composite prepared by this method exhibited elongation about 28% that is much more than conventional W-15%wt Cu composites. This method of production for W–Cu composites has not been reported elsewhere.*

Keywords: *W-Cu composite, Sintering, Infiltration, Submicron Particles, Microstructure.*

1. INTRODUCTION

Tungsten-copper (W-Cu) composites are appropriate candidates in power and nuclear engineering applications such as high-voltage electric contacts, arcing electrodes and deviator plates for fusion reactors, benefiting from the high thermal and electrical conductivity of Cu combined with the wear resistance and refractory characteristic of W[1,2]. To date, the predominant techniques for the production of W-Cu composites are through the infiltration of a tungsten skeleton with copper melt [3-5] and liquid phase sintering [6-8]. Besides, minor addition of nickel as the sintering activator can improve the wetting and adhesion of W and Cu [9-12]. W–Cu composites with high tungsten content i.e. 80-90 wt.% is producible only by infiltration technique[4]. This includes two steps; first preparation of tungsten porous skeleton by pressing and sintering of W powder; second infiltration of molten copper into skeleton [3,4]. Porosity in the part is thus crucially important since it has to be of open type throughout the structure [4,5]. A green compact of tungsten powder with average particle size of 6μm needs a sintering temperature as high as 2150°C to have sintering densification form 60% of theoretical density to 80±2% of that [4]. Below 1900°C usually little densification for tungsten compacts occurs, unless very long sintering time is applied [3-5]. Therefore adopting temperatures lower than 1900°C for sintering the tungsten green compacts implies the use of modified approaches such as applying high consolidation forces, using of very fine powder particles or using the sintering activator i.e. Nickel[9-15]. It was found that a sintering additive which is insoluble in W can segregate to the contact zone between W particles, provide an alloying layer of Ni-W with a eutectic temperature about 1550°C [11]. It can suddenly reduce the performance temperature of W-Cu composite, especially in service [9]. Activated sintering (Ts <2000 °C) resulted in an uncontrolled porous structure as effects such as pore coarsening were observed [3,13]. The sintering temperature can be reduced by using the ultrafine tungsten particles as raw powder[14]. Based on previous studies, tungsten compact with average particle size of 400nm can be sintered to full densities at 1100°C [14]. Despite the high densification, compressibility of ultrafine particles is very limited [14-16]. Also, the porosities between adjacent particles in sintered skeleton will be narrow enough that is not appropriate for copper infiltration [12].

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Conversely, large tungsten particles show good compressibility and desired porosity quality suitable for infiltration [2]. As Ghaderi [4] et.al reported, it is possible to produce tungsten copper composite with a density as high as 17.2 g.cm^{-3} by applying high pressure for compaction of W-particles and using moderate sintering temperature lower than the temperatures used conventionally [4]. However high green density in large particles compacts led to small inter-particle contact area after sintering [16].

It is intended in this study to propose a novel method for fabrication of penetrable porous skeleton of tungsten with desired microstructure and mechanical properties. In other word, this proposed method can help fabrication process of tungsten skeleton without any deleterious effect (such as composition change and formation of closed porosities) on final composite quality.

In this work, W-Cu composite was fabricated by sintering the mixture of micron + submicron powder particles and liquid infiltration routes; the traditional infiltrated W-Cu composite was also prepared for comparison. Microstructure and mechanical properties (i.e. hardness and strength) of these composites were investigated. SEM will be an effective tool in monitoring the porous microstructure in conjunction with quantitative metallography throughout this study.

2. MATERIALS AND METHODS

Tungsten powder with Fisher sub sieve size of 6µm was used as initial powder (Fig. 1a). As mentioned previously, to promote and assist the sintering process of tungsten skeleton, tungsten submicron particles (300nm sizes) were added to initial powders as sintering activator (Fig. 1b). For this purpose, equivalent amount of submicron particles (0, 5, 10 and 20 wt.%) was mixed with tungsten powders by V-blender mixer (Fig. 1c). Mixing of initial powders was done for 24 h at the rate of 60 rpm. Green compacted cylinder having dimensions 50 mm in length and10mm in diameter have been made using cold isostatic press (CIP). Compaction of specimens containing 0, 5, 10 and 20wt.% submicron was carried out at 250MPa. Additionally, specimens containing 20wt.% submicron were compacted at 350, 450 and 550MPa to examine the compressibility of samples. The densities of green compacts have been measured by measuring their volume from their dimensions and measuring the weight by precision electronic balance. Subsequently, compacts containing submicron particles were sintered under hydrogen atmosphere at various temperatures in the range of 1350-1650°C. For a comparison, convectional composite was made by sintering of 6µm tungsten powder at 2000°C, and subsequently infiltration. Oxygen-free copper strips, 99.99 pct purity, were cleaned with sulfuric acid and acetone. They were cut to the calculated weight and were placed in an alumina crucible adjacent of the sintered tungsten compact. Infiltration was carried out at 1300°C for 120 minutes in hydrogen atmosphere. Sintering and infiltration were carried out in tube furnace at the heating rate of 10°C/min. Sintered density was determined using the Archimedes methods according to ASTM B328 standard. Density of infiltrated specimens was measured by Archimedes water immersion method. SEM (Scanning Electron Microscope) in the model of VEGA\\TESCAN and quantitative metallography were used for studying the detailed microstructure of specimens. Comparison is made between the conventional and prepared porous tungsten specimens in this work, in terms of the hardness and tensile strength of the infiltrated composite as well as contiguity. Quantitative metallographic method was used for studying contiguity of tungsten particles in composites. The contiguity (CSS) was calculated using the following Eq. [11].

$$C_{SS} = \frac{2N_{SS}}{2N_{SS} + N_{SL}} \qquad (1)$$

where, N_{SS} and N_{SL} are numbers of solid–solid interfaces and solid–liquid interfaces, respectively.

Following infiltration, hardness measurements were taken using Vickers hardness testing machine at a load and dwell time of 30N and 10s, respectively. Tensile test was done according to ASTME8 and E8 at room temperature by means of INSTRON tensile machine at the rate of 1.25 mm/min.

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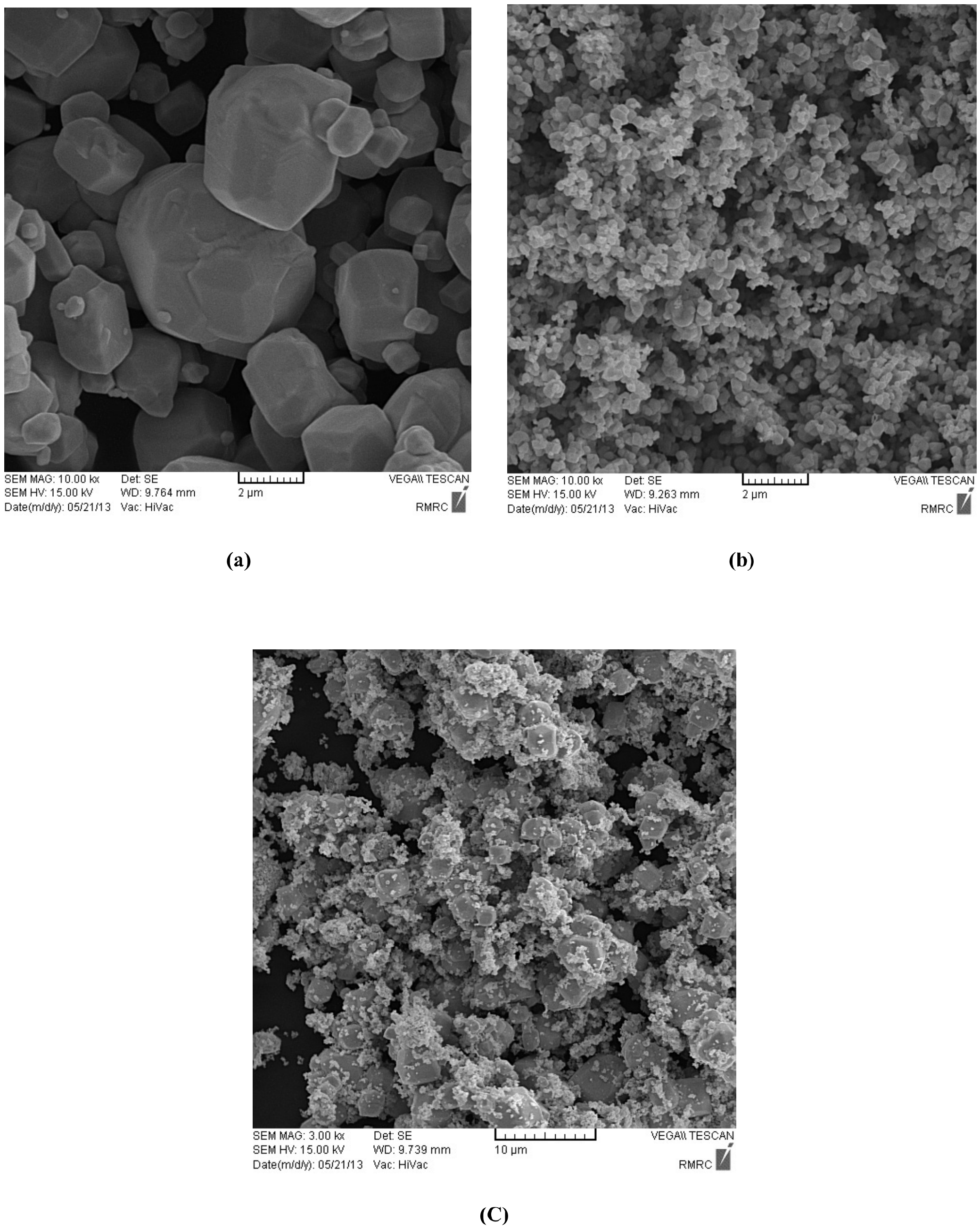

Fig. 1. SEM micrographs of a) 6μm powder particles and b) submicron powder particles and c) mixed powders containing 20wt.% submicron particles.

3. RESULTS AND DISCUSSION

Comparison is made between the submicron particle containing and conventional sintered porous tungsten parts in terms of the compressibility, sinter-ability and microstructure characteristics. Fig. 2a shows the effect of submicron particles on the Green density of compacted specimens at pressure of 250MPa. As illustrated, since the fine particles fill the

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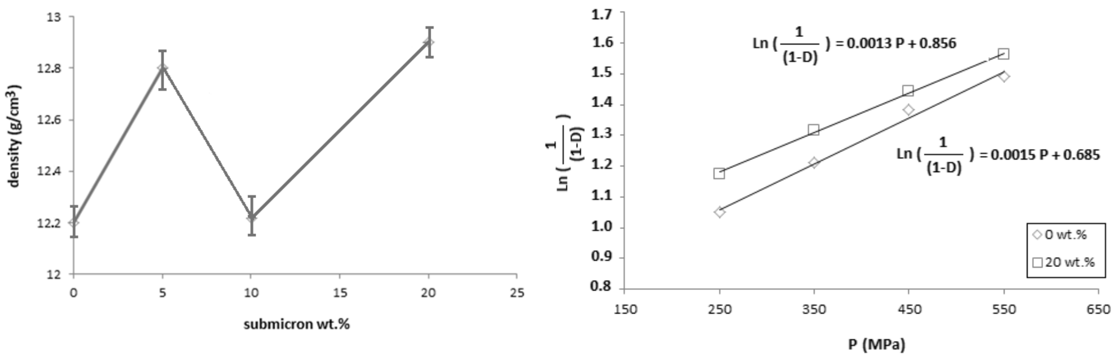

(a) (b)

Fig. 2. (a) density of green specimens after compaction at 250MPa as a function of submicron weight percent, (b) The relation between the compacting pressure and the green density of the compacts containing 0 and 20 wt.% of submicron particles.

interstices between large particles, thus, addition of submicron particles causes higher green density. On the other hand, the presence of 10wt.% of submicron particles in the powder mixture significantly reduces the compact compressibility. This density may be even lower than the density of compacts without submicron particles. In the other word, addition of special amount of submicron particles (10wt.% in this study) to 6μm powder has a negative effect on compressibility of compacts. This result can be explained in terms of the submicron particles extrusion inside inter-connected pores between large particles. The phenomena that occur during consolidation of the powder particles during compaction can be separated into two sequential stages: slip of large particles and submicron particle extrusion into the inter-connected pores between large particles [4]. In the case of 10wt. % submicron particles, these particles place between two adjacent particles and act as an barrier and then increase distance between particles. In other word, large particles are dispersed in submicron particles and reduce the packing density. When the content of submicron additive was increased to 20wt. %, the extra particles intruded in the vacant space between particles and cause higher weight per constant volume. According to Heckel analysis, the rate of change of density increase with compressing pressure for metal powders at any pressure is proportional to the volume fraction of pores at that pressure, depending upon the powder [17]. Heckel described the density-pressure relationship during compressing using an expression of the form:

$$P = \frac{\ln(1-D) - A}{K} \qquad (2)$$

where D is the fraction of theoretical density, P the applied pressure, K proportionality constant related to yield strength of metal and A is a material dependent constant[4, 17]. Using the above Equation for plotting P versus ln[1/(1-D)], The relation between the compacting pressure and the green density of the prepared compacts is shown in Fig. 2b. As a result from Fig. 2b, addition of 20wt.% submicron particles lead to reduction of K that is corresponded to lower compressibility of finer powders. However compacts containing 20wt.% submicron particles show higher green density at all compaction pressures. The results of density measuring of specimens after sintering at 1650°C as a function of submicron particles content are illustrated in Fig. 3. As illustrated, addition of submicron particles has a remarkable influence on the

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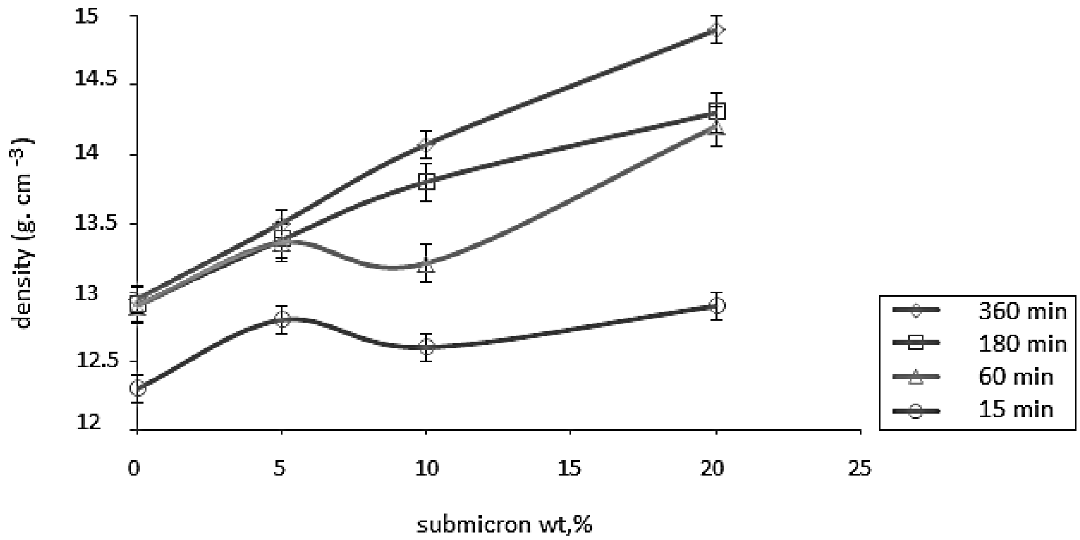

Fig. 3. Density versus submicron particles content for compacts sintered at 1650°C in various times.

densification of tungsten powders. In the case of 0wt.%, increasing sintering time from 60 to 360 min cause no significant change in sintering density. In other words, temperature of 1650°C is not sufficient enough to activate volume or grain boundary diffusion of tungsten atoms. The driving force of diffusion is the minimization of free energy of system, independently from the time [15]. It is also possible to see the increase of the densification with the additions of submicron particles, that couldn't be explained only by increasing of initial porosity of compacts. Addition of submicron particles increases interfacial energy of compact and enhance driving force for sintering. Another reported study in literature found that tungsten powder with a particle size of 400 nm was able to be sintered to over 95% theoretical density at 1100°C within one hour [14]. The powder particle size influences not only the sintering driving force, but also an atomic diffusion path. With respect to sintering of tungsten powder, the dominant mechanism for densification is grain boundary diffusion based on reported studies in literature [13-15]. Powders with finer particle size represent shorter surface diffusion path and then show higher atomic transportation during sintering [15].

Fig. 4 shows SEM micrographs of tensile fracture surfaces for specimens containing 20wt.% submicron particles after sintering for 15min at various temperatures. Fracture was done only to reveal the particles geometry, morphology revolutions and necking growth to investigate densification phenomena at the applied sintering cycle. As illustrated, first phenomenon that can be recognized at 1350°C is the sintering (or connecting) between submicron particles. In fact, due to large free surface energy of submicron particles, necking between adjacent submicron particles takes place at the higher rate than Grain Boundary Structural Transition (GBST)[18] of 6 micron powders. In other words, initial large particles keep their primary polygonal morphology that demonstrated no significant densification of compact. A little change in particles morphology at 1450°C is attributed to initiation of sintering between larger particles. Also, as illustrated, submicron particles begin to be consumed by larger ones in order to reduce total surface free energy of the system. According to microstructural observation, surface atomic diffusion and consuming of submicron particles by large particles are two

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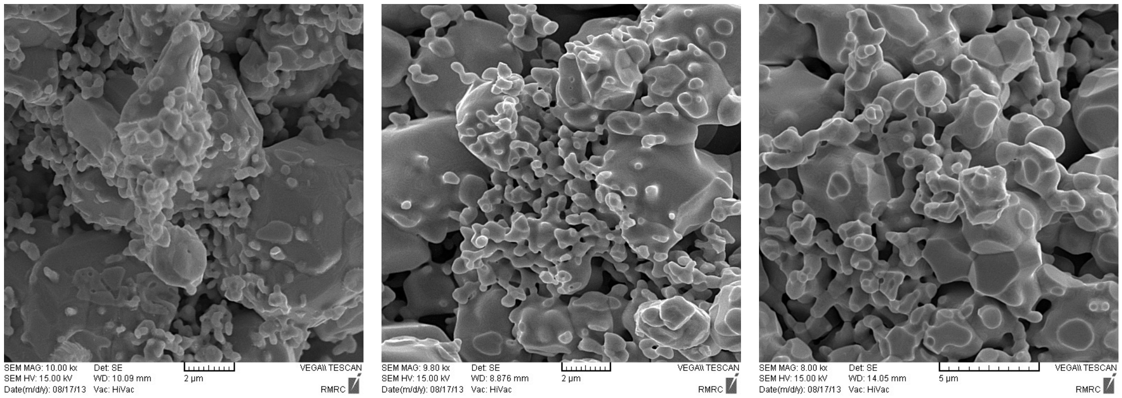
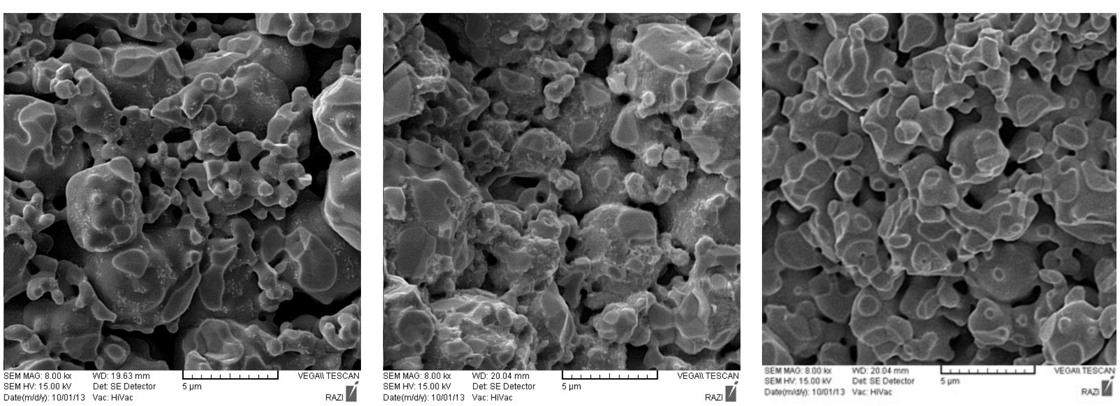


(a) (b) (c)

Fig. 4. SEM micrographs of fracture surfaces of specimens containing 20wt.% submicron particles sintered at (a) 1350, (b)1450 and (c) 1650°C for 15min.

main densification mechanisms in surface activated sintering. This condition continues up to 1650°C until the content of submicron particles decrease due to consuming of submicron particles. Another effect resulted from submicron particles may be rearrangement of large particles due to this consuming mechanism. As discussed above, sintering of mixed powders initiates by sintering of submicron particles. Connections between submicron particles would cause the formation of continuous network between larger particles. Then, inter-connected network of submicron particles acts as a bridge between adjacent large particles and linked them together. When submicron particles begin to be consumed by larger ones, due to formation of this inter- connected network, displacement of large particles takes place subsequently. Fig. 5 shows SEM micrographs of fracture surfaces of specimens containing 20wt.%submicron particles which were sintered at 1650°C for various times.



(a) (b) (c)

Fig. 5. SEM micrographs of fracture surfaces of specimens containing 20wt.% submicron particles sintered at 1650°C for (a) 60min, (b) 180min and (c) 360min.

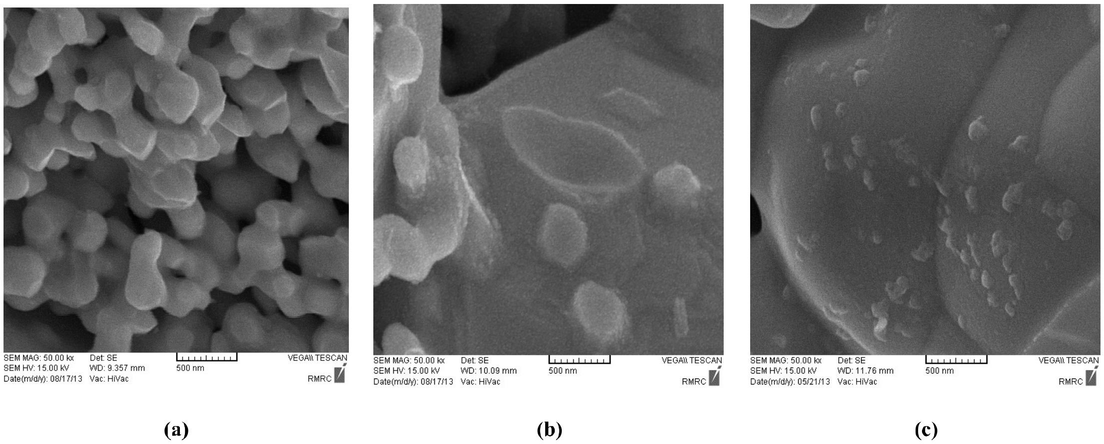


Fig. 6. Fracture surface of 6μm specimen containing 20wt.% submicron particles after sintering to the density of: (a) 12.6, (b) 13.4 and (c) 14.9 g.cm^{-3}. Specimens were sintered at a) 1350°C, (b) 1450°C and (c) 1650°C for 360 min.

As a result from this study, the phenomena that occur during sintering of the mixed powders can be separated into several sequential stages. Fig.6 shows microstructural evolution of 20wt.% containing samples during densification from 12 to 14.9g.cm^{-3}. As illustrated in Fig. 6, These stages consist of: (a) primary sintering of submicron particles at low temperatures (Fig. 6a), (b) formation of inter-connected network between larger particles (Fig. 6b), (c) consuming of submicron particles by larger ones at intermediate temperature and displacement of larger particles and finally solution of remained submicron ones (Fig. 6c). For example, fracture surface of specimen containing 20wt. % submicron particles after sintering at 1650°C for 6h, is shown in Fig. 6c. As illustrated, negligible amount of submicron particles was remained at the surface of initial powders.

Since the aim of this paper is production of W-Cu composite with density of 16±0.2, porous skeletons which sintered to the density of 13.5g.cm^{-3}, were subsequently infiltrated by molten copper at 1300°C. Penetration of liquid through pores of skeleton can be occurred due to capillary pressure as well as high wettability of tungsten particles by liquid. The wetting angle of Cu-melt in the W-Cu system may vary from 8° to 85°[6]. Also, there is no inter-solubility between tungsten and Cu in both solid and liquid state [7, 12]. The results of density measurement of infiltrated composite show efficiency more than 95% can be obtained for specimens containing 20wt.% submicron particles. It show that tungsten- copper composites produced by addition of 20wt.% submicron particles, sintering of the tungsten skeleton at 1650°C and copper infiltration at 1300°C have comparable and even better penetrability than those composites produced conventionally.

SEM micrographs of tensile fracture surfaces of infiltrated specimens with density about 16±0.2g.cm^{-3} containing 0 and 20wt.% of submicron particles are shown in Fig. 7. Remained porosities after infiltration of composite without submicron particles are marked by circle. The primary origin of these porosities can be the small voids formed due to incomplete infiltration or by the rejection of dissolved gases (H_2) during the solidification. These porosities can cause stress concentration at necking regions. Thus, the failure of conventional composite with high content of W usually starts by separation of W/W and develops by producing cleaved tungsten grain after strain hardening the matrix and then matrix rupture occurs [17-19]. In other words, the crack formation in the tungsten composites starts principally through W–W interface rather than through interface between W grain and the matrix phase. Weakness of W-W

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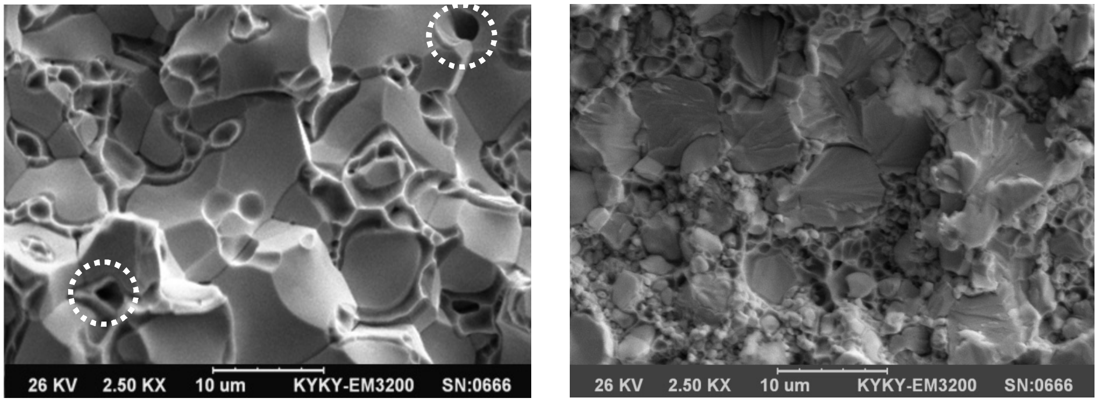

(a) (b)

Fig. 7. BS-SEM micrographs from tensile fracture surface of infiltrated composites with density of 16±0.2 g.cm^{-3} containing: (a) 0wt.% and (b) 20wt.% submicron particles.

interface of traditional infiltrated W-Cu composites may be due to some small impurity phase particles, such as carbides and brittle inter-metallic at the grain boundaries [19-21]. The smooth, often rather round, areas represent the fracture of tungsten grain boundaries. In Fig. 7a, Tungsten particles mainly have demonstrated inter-granular brittle fracture. As shown rather clearly in Fig. 7a, each of the tungsten fractures generates one large dimple and because of the extensive stretching of this surface it appears quite featureless. Several studies of fractography and the fracture mechanisms of W base composites and heavy alloys, show that the failure of alloy with high content of W starts by separation of W/W and develops by producing cleaved tungsten grain after strain hardening the matrix and then matrix rupture occurs. Failure of the strain-hardened matrix around the smaller W grain is evident in these alloys [19-21]. In the case of sample containing 20wt.%submicron particles (Fig. 7b), fracture surface was fully trans-granular type. Trans-granular areas represent the fracture of individual tungsten particlesand show a typical river pattern. The cleavage fractures frequently initiate on many parallel cleavage planes and exhibit twins in the {112} plane and failing like a knife-edge [22]. At 20wt.% submicron particle, the fracture surface of tungsten particles mainly have demonstrated trans-granular brittle fracture.

As an important result, the fracture mode changes from predominantly matrix or W-W interface fracture (in composite without submicron particles) to trans-granular tungsten grain cleavage fracture in the presence of submicron particles. Also, due to mutual dependency of contiguity to W–W contact and to volume fraction of W phase, it seems that contiguity decreases as submicron particle content increase. For specimen without submicron particles (Fig. 7a), W-W interface failure takes place which is associated with the lower strength of W-W interfaces. The absence of interface fracture of the particles (i.e. necks) in submicron particle containing specimens, is an indication of the fact that no significant necking growth take place between large particles and also interfaces between large particles and submicron ones are strong enough to withstand crack propagation. Also, unlike to microstructure in Fig. 7a, no grain growth can be observed in Fig. 7b.

The contiguity, hardness, tensile strength and elongation of infiltrated composite containing 0 and 20wt.% submicron particles are listed in table 1. Tungsten contiguity is proportional to neck size ratio and expresses sintering progress [4, 12]. As shown in table 1, submicron addition produces a supplementary effect on contiguity. It

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Table 1. The contiguity, hardness, tensile strength and elongation of infiltrated composite containing 0 and 20wt.% submicron particles

	(conventional method)	(surface activated sintering)
Submicron particles wt.%	0	20
Density (g cm^{-3})	15.98 ±0.2	16.01 ±0.2
Contiguity (%)	62 ±6	34 ±4
Hardness (VHN)	256 ±2	232 ±3
Tensile strength (MPa)	504 ±6	510 ±10
Tensile elongation (%)	15 ±3	28 ±2

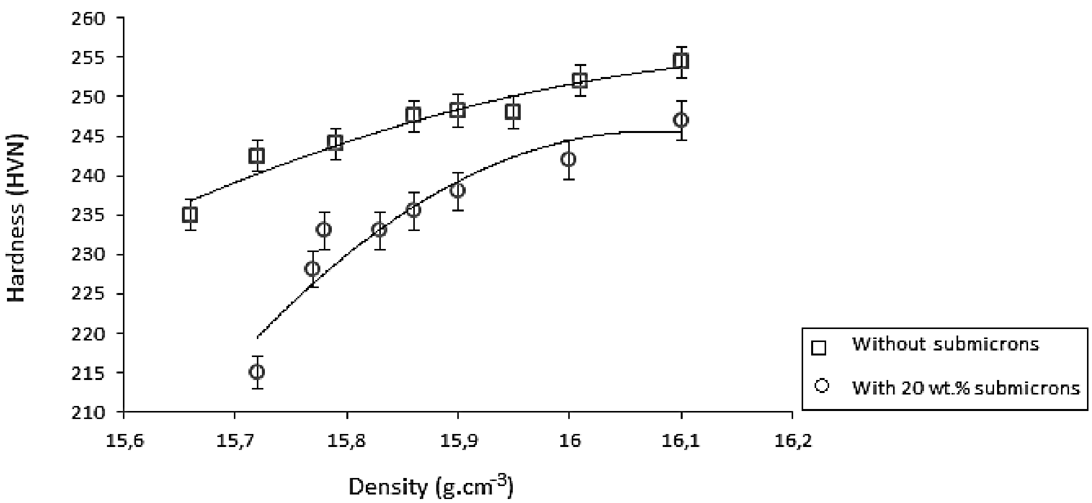

Fig. 8. Comparison between hardness of specimens containing 0 and 20 wt.% submicron particles. Specimens with submicron particles show lower hardness.

has been reported that, the mechanical properties of W–Cu composites would be affected by microstructural parameters such as the W–W contiguity [10, 23-25]. In the case of 20wt.% submicron particle, tensile strength was obtained 510MPa. Compared with sample without submicron particles (traditional composites), composite prepared by 20wt.% fine particles exhibits rather same strength and better ductility under tensile condition. This result is mostly due to the lower contiguity microstructure of the W grains compared to the conventional W-Cu composite. The analysis of the failure mechanism discussed previously and data listed in Table 1 shows that W-W contiguity evidently decreases due to addition of submicron particles, and then, metallurgical bonding is formed between W and the matrix instead of W-W interfaces. With addition of submicron particles, interconnection of Cu phase as well as interfacial area between W and Cu was frequently observable and hence W-W contiguity decreased. All these above factors are beneficial to increase the ductility. Meanwhile, the decrease of W-W contiguity and the increase of the interfacial strength account for the ductility increase under tensile condition. Several studies of W-Cu composites, verify that the porosity sites and solid-solid contacts are the weak point of W-Cu compacts [10, 21]. Based on table 1, the hardness of W-Cu composite is proportionally related to W-W contiguity.

Relation between hardness and addition of

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submicron particles as a function of composite density is shown in Fig. 8. It can be found that the Vickers hardness of the W-Cu composites containing 20 wt.% submicron particles is much lower than that of the conventional W-Cu for all composite densities. This result is thought to result from the lower contiguity structure of the W grains compared to the conventional W-Cu composites due to the smaller necking area between large particles in presence of fine particles. As mentioned, the higher ductility property in this high density W-Cu composite is expected because of the connectivity of copper phase due to lower contiguity.

4. CONCLUSIONS

In summary, novel W-Cu composites with submicron particles have been fabricated by sintering and liquid infiltration routes. The microstructure evolution during sintering of the W powder mixtures was investigated for the purpose of developing a desired porous skeleton. It was found that the W powders containing 20wt. % submicron particles during sintering at 1650°C reached a density of 13.5g.cm^{-3} with a homogeneous microstructure. Microstructure observation revealed that the present high density W-Cu composites (16g.cm^{-3}) consist of low contiguity structures of W-W grains and an interconnected Cu phase located around the W grains. It was found that:

1. The results of this research showed that it is possible to produce Tungsten-copper composite with a density as high as 16 g.cm^{-3} by addition of submicron particles to 6μm W powders and using moderate sintering temperature lower than the temperatures used conventionally. By this method, high temperature (e.g. 2200°C) would not be required for sintering of tungsten compacts.
2. The phenomena that occurred during densification of the mixed powder during surface activated sintering may be separated into several sequential stages: sintering of submicron particles at low temperatures, formation of connected network of submicron particles between larger powders, consuming of submicron particles by larger ones at intermediate temperature and displacement of large particles.
3. Composites prepared by this new method exhibited better ductility and rather similar tensile strength compared to conventional composites. Tensile strength and ductility were obtained about 510MPa and 28%, respectively.
4. The Vickers hardness of the sintered W-15wt.% Cu compacts containing 20wt% submicron particles was measured about 232Hv, a value much lower than that of the conventional similar W-Cu composite (256Hv). This result was mostly due to the lower contiguity microstructure of the W grains compared to the conventional W-Cu composite.
5. Although this method could replace the conventional process, there is still a potential interest in optimizing the current process to make more uniform porous tungsten parts which possess the least amount of scattering in their characteristics.

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Conflict of Interest: The authors declare that they have no conflict of interest.

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