Journal of Alloys and Compounds 505 (2010) 510–515

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Spark plasma sintering of mechanically alloyed in situ copper–niobium carbide composite

B.D. Long [a,b], R. Othman [a], M. Umemoto [b], H. Zuhailawati [a,*]

[a] School of Materials and Mineral Resources Engineering, Engineering Campus, Universiti Sains Malaysia, 14300 Nibong Tebal, Penang, Malaysia
[b] Department of Production Systems Engineering, Toyohashi University of Technology, Japan

ARTICLE INFO

Article history:
Received 26 March 2010
Received in revised form 20 June 2010
Accepted 24 June 2010
Available online 3 July 2010

Keywords:
Metal matrix composites
Mechanical alloying
Hardness
Electrical conductivity
Spark plasma sintering

ABSTRACT

A Cu–NbC composite with high electrical conductivity and high microhardness was synthesized by mechanical alloying and densified using spark plasma sintering (SPS). Mixtures of Cu–NbC powders corresponding to volume fractions of 1, 5, 15 and 25 vol% NbC were milled in a high energy planetary mill under argon atmosphere for 30 h using ethanol as process control agent. The Cu–NbC as-milled powder was sintered using spark plasma sintering temperatures between 900 and 1000 °C. X-ray diffraction investigation showed that NbC started to form in the copper matrix during ball milling and the reaction between Nb and C was completed after 10 min of SPS sintering. Electrical conductivity and density of the Cu–15 vol% NbC composite increased with increasing sintering temperature. The results showed the superior properties of SPS-prepared Cu–NbC composite: electrical conductivity is almost 4 times higher and microhardness is 3.5 times higher than with normal sintering. A highest density of 98% and electrical conductivity of 45.6% IACS were obtained in the Cu–1 vol% NbC composite. The highest microhardness of 452.9 Hv was achieved in the Cu–25 vol% NbC composite.

1. Introduction

Every year demand increases for materials which have high electrical conductivity and high mechanical strength, such as copper-based alloys [1]. However, copper-based alloys like brasses and bronzes have suffered from problems like dezincification and inferior elevated temperature properties as well as less stable microconstituents [2]. A composite of copper matrix reinforced with NbC particle composite with high electrical conductivity, good mechanical strength and thermal stability has been found to solve such problems and therefore able to meet the demand. Copper-based metal matrix composites reinforced with hard phase particles of carbides, oxides, and borides have become an attractive area of research [3–8]. Copper composites have a wide range of applications in the electronics field such as for production of electrodes for electrical spot welding, sliding electrical contacts and high-performance switches [1,9–11], enhancement of thermal conductivity such as heat exchanger, and in actively cooled components [12] and the wear industry [4,5,13].

Mechanical alloying (MA) is a solid state powder processing method which involves repeated cold welding and fracture of particles as a result of high energy ball powder collisions. MA is an appropriate technique for the development of metal matrix composite (MMC) materials reinforced with in situ strengthening precipitates with better properties [14]. Through this process, a very fine, nano-scale, homogeneous distribution of reinforcement particles through the copper matrix is achieved.

It is a challenging task to identify an appropriate method and suitable processing conditions for sintering nanometric powders obtained by mechanical alloying that will produce bulk bodies with desirable and superior properties while still preserving the nanostructure. In principle, a few sintering methods have been used such as vacuum sintering, hot pressing, hot isostatic pressing (HIP), shock consolidation, pulsed current activated sintering (PCAS), microwave sintering, high-frequency induction sintering and the newly developed method of spark plasma sintering (SPS) [15,16]. Conventional sintering routes such as hot consolidation require higher temperature and longer sintering time, which lead to grain growth and high porosity [17–19]. SPS, a field-activated sintering technique, is a suitable consolidation technique for sintering as-milled Cu–NbC powder. SPS can be used with high heating and cooling rates thus shortening processing time. The powder sample can achieve full density and grain growth can be avoided as well [20]. See [21,22] for more detailed discussion of SPS.

Cu–NbC composite has been synthesized by mechanical alloying in previous research [1,3,7,11]. However, in this research, the as-milled powders were cold compacted and then sintered by normal sintering, which can cause the resulting product to be lower in

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[*] Corresponding author. Tel.: +60 4 5995285; fax: +60 4 5941011.
E-mail address: zuhaila@eng.usm.my (H. Zuhailawati).

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doi:10.1016/j.jallcom.2010.06.150

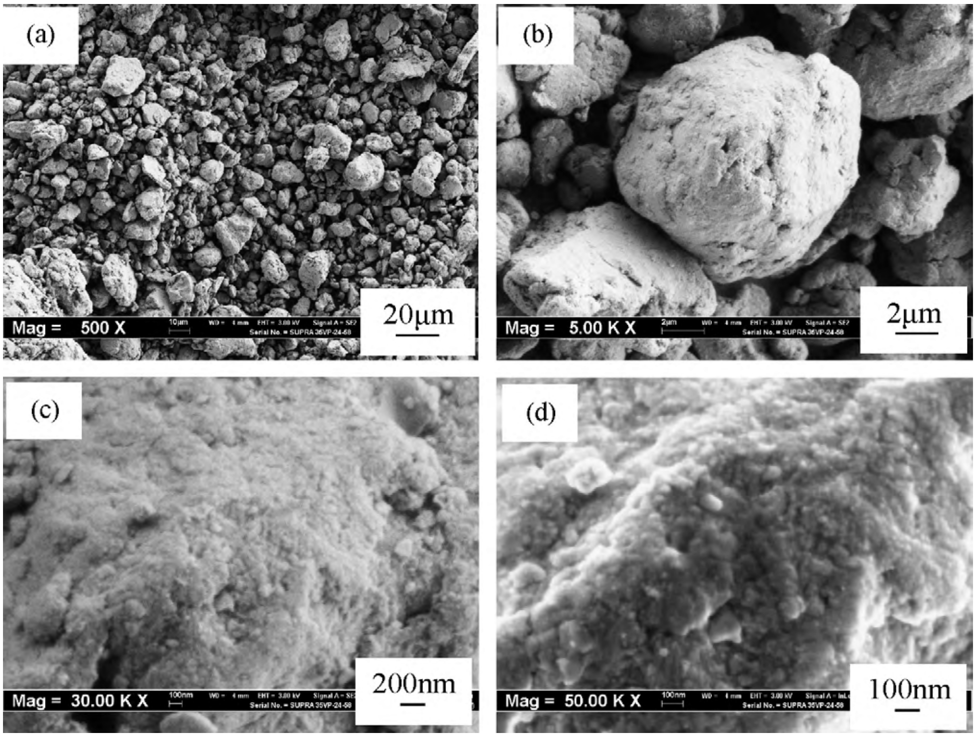

Fig. 1. SEM morphology micrograph of as-milled Cu–15 vol% powder at different magnifications of: (a) 500, (b) 5000, (c) 30,000 and (d) 50,000×.

density and have coarse particle size. Furthermore, we lack information about the relationship between electrical conductivity and microhardness or that between electrical conductivity and volume fraction of the reinforcement.

The aim of this research, therefore, is to produce via mechanical alloying and spark plasma sintering a Cu–NbC composite which has high electrical conductivity in combination with high microhardness. In order to study the correlation between properties and processing parameters, mixtures of Cu, Nb and C powders corresponding to different volume fractions of 1, 5, 15 and 25 vol% NbC were subjected to mechanical alloying. As-milled Cu–15 vol% NbC powders were sintered using SPS at temperatures in the range of 900–1000 °C for 15 min at a pressure of 100 MPa in order to study the effect of sintering temperature on the properties of Cu–NbC composite. The best sintering conditions for powders of this volume fraction were then applied for sintering the other as-milled powders (of Cu–1, 5 and 25 vol% NbC).

2. Experimental procedures

Cu, Nb and C powders were used to prepare powder mixtures with compositions of 1, 5, 15 and 25 vol% NbC. Cu powder was of 99.9 purity with particle size $\leq$180 μm; Nb powder was of 99.5% purity with particle size $\leq$7 μm; and C powder was of 99.9% purity with particle size $\leq$10 μm.

In order to prevent oxidation of the powders, loading and discharging of the powders was carried out inside an argon filled-glove box. The mixture powders were milled using a Fritsch “Pulveristte 5” planetary ball mill with two steel containers of 500 ml at a rotation speed of 250 rpm. The containers were sealed under an Ar atmosphere to prevent oxidation of the powders. Ethanol was used as a process control agent in order to prevent excessive cold welding of these powders during ball milling. The SUJ-2 balls which have high toughness and wear resistance were chosen in order to reduce contamination during ball collisions. The balls used were 10 mm in diameter and powder-to-ball weight ratio was fixed at 1:10.

As-milled Cu–NbC powder with a composition corresponding to 15 vol% NbC was sintered at 900, 950 and 1000 °C for 15 min to investigate the effect of sintering temperature on properties of the Cu–NbC composite. In order to study the effect of NbC on mechanical and electrical properties of Cu–NbC composite, the as-milled powders with compositions corresponding to 1, 5, 15 and 25 vol% NbC were sintered at 1000 °C for 10 min. The in situ chemical reaction and consolidation of the milled powders was performed using Dr. Sintering® 2040 spark plasma sintering (SPS) system (Sumitomo Coal Mining, Tokyo, Japan). The SPS processes were conducted with applied pressure of 100 MPa during sintering in vacuum atmosphere of 5 Pa. In order to compare the results of this process with those of normal sintering, as-milled Cu–15 vol% NbC powder was cold compacted and then sintered in a tube furnace at 1000 °C for 1 h under an argon atmosphere.

The as-milled powders and sintered samples were characterized using X-ray diffraction patterns (XRD) for phase identification. XRD analyses were carried out with a RIGAKU RINT-2500 X-ray diffractometer using Cu-Kα radiation. Microstructure of the composites was investigated using scanning electron microscopy (SEM). Hardness and electrical conductivity were measured using Vicker's microhardness tester and Ulvac ZEM 1 electrical conductivity equipment, respectively, with sample size of 1.5 mm × 3 mm × 17 mm. For comparison purposes, a non-deformed Cu plate of 99.96% purity was also subjected to microhardness measurement. Electrical conductivity was measured in IACS (International Annealed Copper Standard) unit where 100% IACS is equal to 58.0 $\mathrm{m}/\Omega\,\mathrm{mm}^2$. The density of sintered samples was measured by Archimedes' principle.

3. Results and discussion

3.1. The formation and distribution of NbC in the copper matrix

The formation of NbC in the copper matrix was studied using various volume fractions of NbC (1, 5, 15 and 25 vol%). Mixtures of Cu, Nb and C powders were milled for 30 h in argon. The morphology of the as-milled powder was examined by SEM, as shown in Fig. 1. The as-milled powder particles are distributed with different range of sizes, as shown in Fig. 1(a), with average particle size of 16 μm. In order to understand the origin of the coarse particles in the as-milled powder particle after mechanical alloying, the as-milled powder was observed under SEM with higher magnification, as shown in Fig. 1(b)–(d). It can be seen that after mechanical milling the coarse particles appeared to be agglomerates of finer particles.

Fig. 2 shows the in situ formation of NbC in the copper matrix after ball milling. According to the results, no Nb or NbC peaks were observed in the as-milled Cu–1 and 5 vol% NbC after milling for 30 h since both as-milled powders had low Nb and C concentrations, as shown in Fig. 2(a) and (b). XRD only detected NbC phase in the as-milled powder containing 15 vol% NbC and 25 vol% NbC, as shown in Fig. 2(c) and (d). This finding indicates that the impact energy

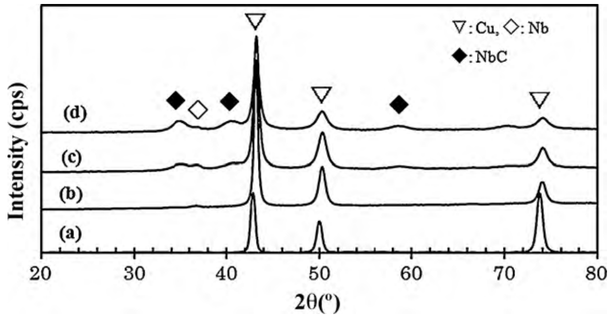

Fig. 2. XRD patterns of as-milled powders with different volume fractions of NbC: (a) 1, (b) 5, (c) 15 and (d) 25 vol% NbC.

during ball milling is sufficient to activate the reaction between C and Nb to form to NbC precipitate. This explanation is supported by the equilibrium thermodynamic data of Gibbs free energy for NbC formation, ΔG_f is equal to $-137\,\text{kJ}\,\text{mol}^{-1}$ at 300 K, indicating that it is thermodynamically feasible for the reaction between Nb and C to take place at room temperature. In situ formation of NbC in the copper matrix was also investigated by [11,23] who noted that the XRD peak of NbC was broadened due to the formation of very fine NbC particles and high strain after mechanical alloying. In Fig. 2, the Nb peak is also observed, indicating residual Nb due to incomplete NbC formation.

Fig. 3(a) and (b) are high-magnification SEM micrograph showing the distribution of NbC in the as-milled Cu–15 vol% NbC powder and the corresponding EDX point chemical analysis. It can be clearly seen that very fine NbC particles with average particle size of 179 nm are homogeneously distributed in the copper matrix after ball milling for 30 h. The EDX point of copper matrix, as marked by a white cross in Fig. 3(a), is shown in Fig. 3(c).

3.2. Varying sintering temperatures

In order to study the effect of sintering temperature on properties of the composite, as-milled Cu–15 vol% NbC powders were sintered at different temperatures with applied pressure of 100 MPa and heating rate of 100 °C/min for 15 min. The formation of NbC in the copper matrix was confirmed by XRD in Fig. 4. It can be seen that the intensity of the NbC XRD peaks increased with increasing sintering temperature due to the increasing formation of NbC and coarsening of particle size [24].

The density and electrical conductivity of the composite was found to increase with sintering at higher temperature and their maximum values were obtained at a sintering temperature of 1000 °C, as shown in Fig. 5(a). The increase of electrical conductivity is mainly attributed to an increase in density, and minor effects are mainly attributed to recrystallization and stress relief and the elimination of dislocation since the composite was sintered at high temperature [25,26]. It is also important to note that the increasing precipitation of NbC in the copper matrix at higher sintering temperature causes the restoration of the distorted lattices, reducing the impurity scattering, hence resulting in an increase in electrical conductivity [27]. Microhardness of the composite

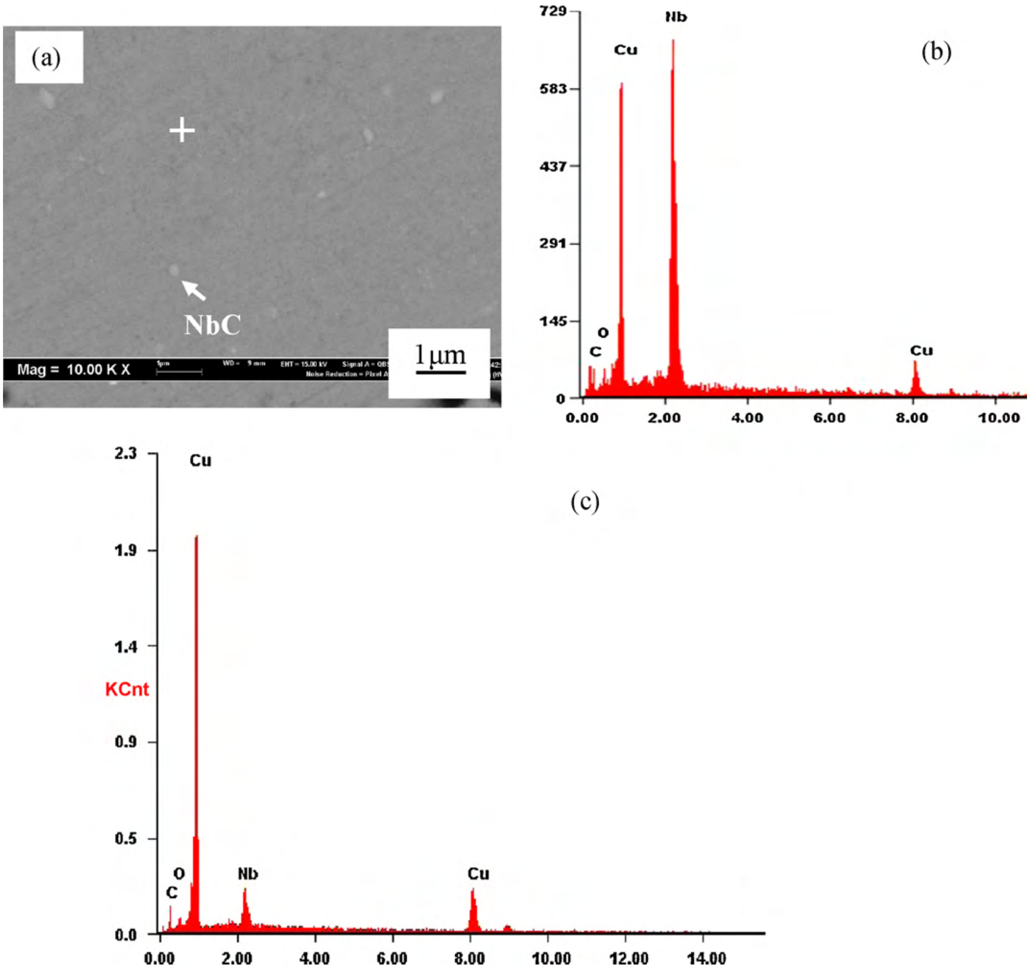

Fig. 3. (a) Distribution of NbC in the copper matrix of as-milled Cu–15 vol% NbC and EDX spectrum of (b) NbC component and (c) copper matrix.

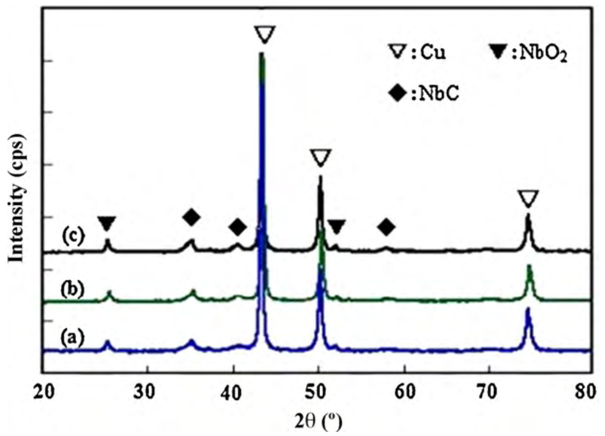

Fig. 4. XRD patterns of as-milled Cu–15 vol% NbC powder after sintering temperatures of: (a) 900 °C, (b) 950 °C and (c) 1000 °C for 15 min.

increased with increasing sintering temperature and reaches the maximum value of 342 Hv after sintering at 950 °C, as shown in Fig. 5(b). Microhardness then decreased when the composite was sintered at 1000 °C due to higher occurrence of recrystallization [24].

3.3. Consolidation of variation in as-milled Cu–NbC powders

In order to obtain the Cu–NbC composite with high density and high electrical conductivity, as-milled Cu–NbC powders corresponding to Cu–1 to 15 vol% NbC were sintered at 1000 °C under applied pressure of 100 MPa. The sintering soaking duration was reduced to 10 min in order to maintain high microhardness. The XRD patterns of Cu–NbC composites after sintering are shown in Fig. 6, which shows that the intensity of NbC peaks increased with increasing volume fraction of NbC. It can be seen that Nb disappeared and the formation of NbC was completed just after sintering for 10 min.

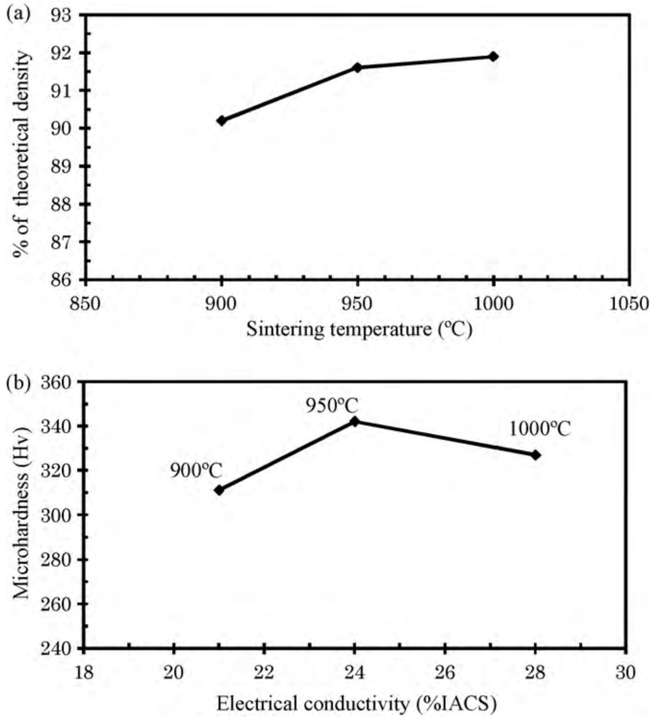

Fig. 5. Properties of Cu–15 vol% NbC composite with different sintering temperatures: (a) density of the composites and (b) electrical conductivity versus microhardness.

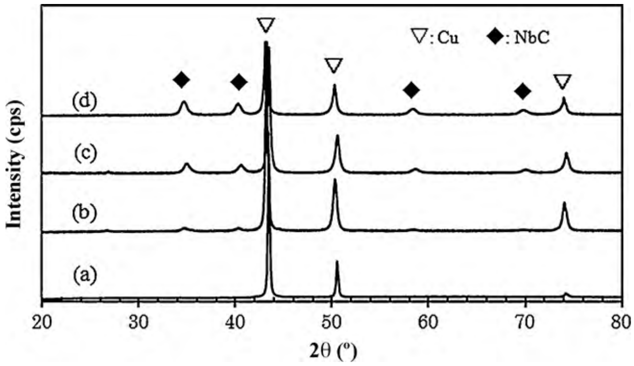

Fig. 6. XRD patterns of sintered Cu–NbC composite with (a) 1, (b) 5, (c) 15 and (d) 25 vol% NbC.

After sintering, the maximum density value was achieved in Cu–1 vol% NbC composite with 98.0% of theoretical density. The densities of other composites were around 97.0% of theoretical density, as shown in Table 1. A typical SEM observation of NbC in the consolidated sample without etching, shown in Fig. 7, reveals good bonding between NbC and the copper matrix. This result indicates an advantage of an in situ method over an ex situ method, since the reaction between reinforcement and matrix observed in the ex situ method resulted in decreased microhardness and tensile strength [9,28].

3.4. Effect of NbC volume fraction on microhardness

The relationship between microhardness and volume fraction of NbC is shown in Fig. 8. Microhardness of the composite was shown to gradually increase with increasing volume fraction of NbC. The maximum microhardness of the composite was 452.9 Hv in a Cu–25 vol% NbC composite.

With the addition of 1 vol% NbC, the microhardness of a Cu–NbC composite (162.0 Hv) was observed to be more than 3.6 times higher than that of non-deformed Cu (45 Hv). The increased microhardness of the Cu–NbC composites over than that of pure copper may be due to the composites' incorporation of NbC hard phase. In a metal matrix composite material, the reinforcement phase prevents plastic deformation by blocking the dislocation movement in the matrix phase under mechanical loading [29,30]. In Cu–NbC composite, the copper matrix was first deformed upon applying the load, whilst NbC acted as obstacles to the moving dislocations in the copper matrix, leading to an increase in microhardness of the composite. With higher volume fraction of NbC, the resistance to the motion of dislocations is also increased, and led to an increase in microhardness.

3.5. Effect of NbC volume fraction on electrical conductivity

In order to study the effect of volume fractions of NbC on the electrical conductivity of the composite, the electrical conductivity was plotted versus volume fraction of NbC, as shown in Fig. 9. Electrical conductivity of the composite gradually decreased with increasing volume fraction of NbC. The maximum electrical conductivity of 45.5% IACS was obtained in the Cu–1 vol% NbC composite. The electrical conductivity gradually decreased, reaching a minimum value of 18.6% IACS when 25 vol% NbC was used as the reinforcement.

The electrical conductivity of any two-phase composites is determined by the electrical conductivities of the constituent phases, the volume fractions, and the distribution of the constituent

Table 1
Density of Cu–NbC composites after sintering by SPS.

Composite	Cu–1 vol% NbC	Cu–5 vol% NbC	Cu–15 vol% NbC	Cu–25 vol% NbC
Density (g/cm^3)	8.8	8.7	8.5	8.4
Theoretical density (%)	98.1	97.0	96.8	97.0

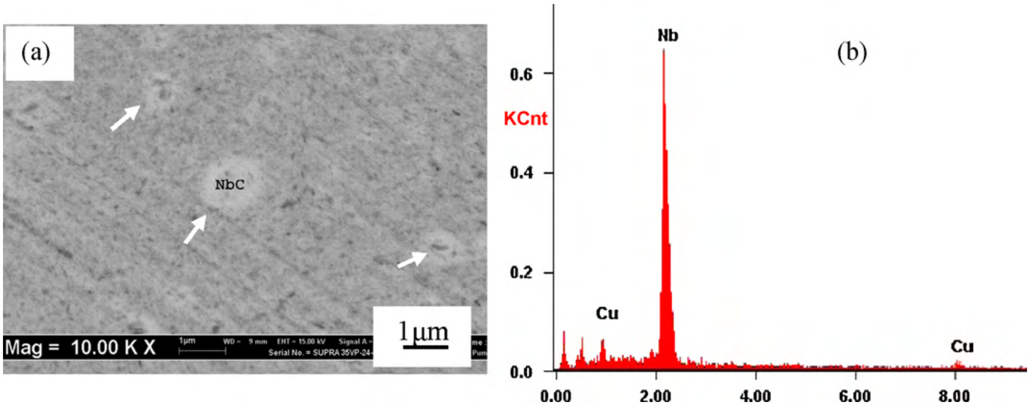

Fig. 7. (a) A typical NbC in the Cu–15 vol% NbC composite after sintering and (b) EDX spectrum of NbC particle in the copper matrix.

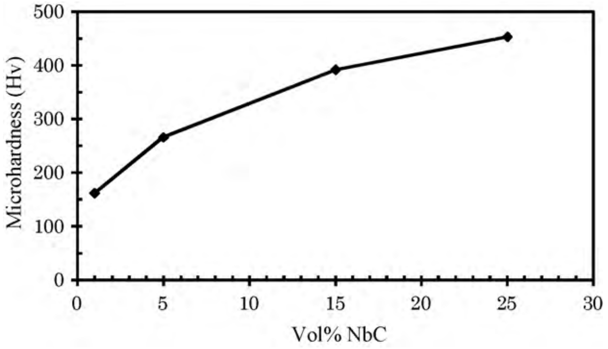

Fig. 8. Microhardness versus volume fraction of Cu–NbC composites.

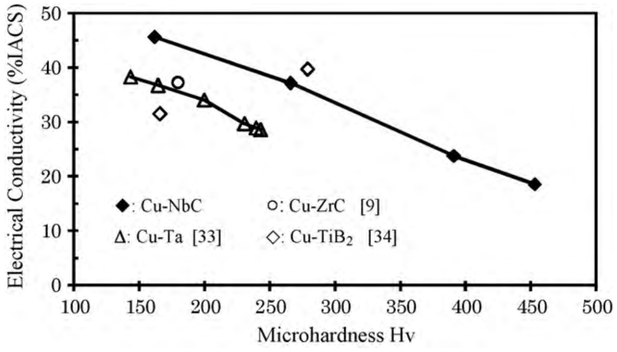

Fig. 10. Correlation of electrical conductivity with microhardness and comparison of properties of present Cu–NbC composites with other copper composites from the literature.

phases, furthermore by the size, shape, orientation and spacing of the phases, and finally, by interaction between phases and the preparation method [31]. However, in the present work, the electrical conductivity of the Cu–NbC composite appears to be determined mainly by the volume fraction of NbC. With increasing amount of NbC reinforcement, the mean free conductive path increases, resulting in a decrease in electrical conductivity. In addition, the increased NbC volume fraction may account for the decrease in Cu grain size and the resultant increase in grain boundary volume [32]. The increasing grain boundary volume led to increased barriers to the conductive path, which can also result in lower electrical conductivity [33].

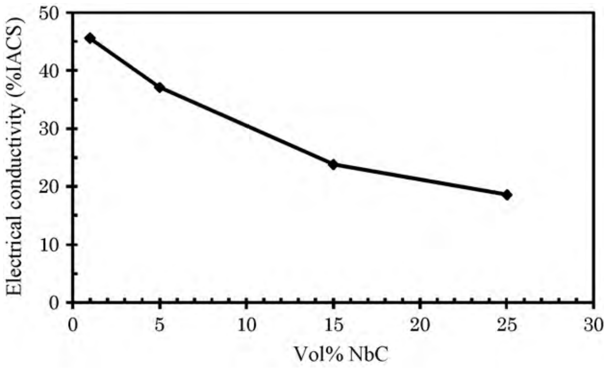

Fig. 9. Electrical conductivity versus volume fraction of Cu–NbC composites.

3.6. *Correlation of electrical conductivity and microhardness*

In applications of copper-based materials, it is typically required that the material have a good combination of electrical conductivity and microhardness or high strength. Therefore, it is necessary to determine the relationship between the electrical conductivity and the microhardness of the Cu–NbC composites. The correlation between electrical conductivity and microhardness of the Cu–NbC composites is plotted in Fig. 10. Findings of other researchers on the electrical conductivity and microhardness of other copper composites prepared by ball milling (Cu–ZrC [9], Cu–Ta [33] and Cu–TiB$_2$ [34]) are also plotted in Fig. 9. It can be seen that the microhardness is inversely proportional to their electrical conductivity. A similar relationship is also observed in the case of the Cu–Ta composite.

It can be seen that the electrical conductivity of Cu–NbC composite in the present work is much higher than that reported for the Cu–Ta and Cu–TiB$_2$ composites. According to that study, electrical conductivity of the Cu–1 wt% TiB$_2$ composite was 35.1% IACS after sintering and increased to 41.5% IASC only after rolling. In the present work, Cu–1 vol% NbC evidenced higher electrical conductivity without a secondary process such as rolling.

3.7. Comparison to normal sintering

The formation of NbC in the copper matrix with high densification occurs in a short time during SPS, showing the advantage of SPS over other sintering processes such as normal sintering, hot pressing (which requires higher pressure), higher sintering temperatures and longer sintering times of usually 1–2 h. It has also been reported that porosity is lower for samples densified using SPS [9,17]. In other studies [1,35], 1 h sintering was found to be required to complete the formation of NbC in the copper matrix by normal sintering whilst the present work found that the reaction of Nb and C was completed after sintering for only 10 min using SPS. Unfortunately, detailed data from those other studies on the properties of Cu–NbC composite such as density, electrical conductivity and the relationship between electrical conductivity and mirohardness are not available, thus precluding further comparison of the properties of the resultant composites.

To allow comparison of the properties of composites treated by SPS with those treated with normal sintering, as-milled Cu–15 vol% NbC powder was compacted at 500 MPa and sintered normally at 1000 °C in Ar atmosphere for 1 h. The results show that the composite had 81.0% relative theoretical density, electrical conductivity of 4.1% IACS and microhardness of 114.1 Hv compared to the composite with the same composition that had been sintered using the novel SPS method, with 96.8% relative theoretical density, electrical conductivity of 23.8% IACS and microhardness of 391.5 Hv. The properties of SPS-prepared Cu–NbC composite are clearly superior to those of normally sintered Cu–NbC composites: due to the higher density of the SPS-treated composites, their electrical conductivity is almost 4 times higher and their microhardness is 3.5 times higher than those of composites treated with normal sintering.

The composites developed in this research can be applied in the field of electronic technology, where devices require a combination of high electrical conductivity with high strength. Possible applications of Cu–NbC composites could be as electrodes for electrical spot welding, as relay blades and contact supports and as, sliding electrical contacts [9]. In this research, Cu–5, 15, and 25 vol% NbC composites showed very high microhardness, even with sintering at the high temperature of 1000 °C. Even in the case of Cu–1 vol% NbC composite, microhardness was also high. This shows that the Cu–NbC composite has thermal stability at elevated temperatures. Compared to other copper-based alloys (e.g. Cu–Cr, Cu–Fe-P), the Cu–NbC composites had lower electrical conductivity but strength of the other alloys drastically dropped at temperatures above 500 °C due to the coarsening of the reinforcement particles [9,27,36]. The Cu–NbC composites were found to be appropriate for applications in fields, requiring both high electrical conductivity and high microhardness or high strength at elevated temperature.

4. Conclusions

NbC was formed in situ in the copper matrix after ball milling for 30 h. No NbC was detected in the samples reinforced with 1 and 5 vol% NbC due insufficient volume fraction of NbC. The formation of NbC in the copper matrix was complete after sintering by SPS for 10 min. The electrical conductivity and density of Cu–15 vol% NbC composites significantly increased with increasing sintering temperature, mainly due to their increasing density, with minor influences of recrystallization, stress relief, and the elimination of dislocation. A high density (>97%) was obtained in the Cu–NbC composites after sintering at 1000 °C.

Electrical conductivity of the Cu–NbC composites varied inversely with microhardness. The increase in electrical conductivity was of almost the same magnitude as the decrease in microhardness with reducing volume fraction of NbC reinforcement. The maximum electrical conductivity value of 45.6% IACS was obtained in the Cu–1 vol% NbC composite, whilst its microhardness was 162 Hv. The maximum microhardness value of 452.9 Hv was achieved in the Cu–25 vol% NbC composite, whilst its electrical conductivity is 18.6% IACS. Good thermal stability was observed in the Cu–NbC composite.

Acknowledgements

This work was supported by The Japan International Cooperation Agency (JICA) under the Asia University Network/Southeast Asia Engineering Education Development Network (AUN-SEED/Net) and Science Fund Grant provided by the Ministry of Science, Technology and Innovation, Malaysia (Project No. 03-01-05-SF0374). The authors would like to thank Dr. Yoshikazu Todaka for his advice.

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