Research Article

Comparison of Two Powder Processing Techniques on the Properties of Cu-NbC Composites

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An *in situ* Cu-NbC composite was successfully synthesized from Cu, Nb, and C powders using ball milling and high pressure torsion (HPT) techniques. The novelty of the new approach, HPT, is the combination of high compaction pressure and large shear strain to simultaneously refine, synthesize, and consolidate composite powders at room temperature. The HPTed Cu-NbC composite was formed within a short duration of 20 min without Fe contamination from the HPT's die. High porosity of 3–9%, Fe and niobium oxidations, from grinding media and ethanol during ball milling led to low electrical conductivity of the milled Cu-NbC composite. The electrical conductivity of the HPTed Cu-NbC composite showed a value 50% higher than that of milled Cu-NbC composite of the same composition.

1. Introduction

Powder metallurgy (P/M) is the most common method for the synthesis of metal matrix composites (MMCs) [1–3], whereby the refinement and consolidation are the most important parts of this process. The refinement of powder particles leads to increasing mechanical strength of MMCs as given by the Hall-Petch relationship [4], as well as conferring excellent consolidation/high apparent density which enhances the properties of MMCs [5]. In addition, P/M shows many other advantages in the synthesis of MMCs such as homogeneous distribution of reinforcement in the matrix, fine grain size, synthesis process at low temperature, and cost saving. The excellent physical and mechanical properties of MMCs, in particular Cu-NbC composites, are applied widely in electrical fields such as electrodes for spot welding, contact materials in high power switches, sliding electrical contacts, and thermal management devices.

In P/M, ball milling (BM) is well known as a powerful method to synthesize and refine MMCs with extremely fine and homogenous distribution of reinforcement in a metal matrix [6, 7]. However, BM imposes some disadvantages such as contamination from the grinding media and the process control agents (PCAs) during milling [6].

Cold compaction or cold isostatic pressing was used as a conventional consolidation method followed by sintering [8, 9]. However, these consolidation methods have a major problem which relates to a low density of the bulk material [9, 10]. Therefore, a second process such as hot extrusion, rolling, or forging was usually needed to completely consolidate the final product [11–13]. Previous research work [14] showed that increasing the density of Cu-NbC composite from 81 to 96.9% led to an increase in hardness and electrical conductivity by 3.4 and 5.8 times, respectively. In addition, the consolidation process becomes much more difficult when hard milled powders [15, 16] and composite powders containing high volume fraction of reinforcement [5, 17, 18] are to be consolidated.

Various approaches had been developed to improve the density of the final product by applying high temperature and pressure during sintering, that is, hot pressing or hot pressing followed by rolling [17, 19, 20]. However, powders held at elevated sintering temperatures for long duration

lead to grain growth and subsequent loss of mechanical properties [21]. Spark plasma sintering (SPS) and shock compaction with very high heating/cooling rates [14, 22–24] or extremely high pressure hot pressing (7.7 GPa) [25] have been applied to achieve full density composites without significant grain growth. These consolidation methods are expensive and difficult to apply. In recent years, severe plastic deformation (SPD) methods such as equal channel angular pressing (ECAP) [5, 26], torsion extrusion (TE) [27], and, especially, high pressure torsion (HPT) [28–32] seem to be promising methods for the consolidation of powders.

In this research work, and for the first time, HPT was used to synthesize *in situ* Cu-NbC composite from Cu, Nb, and C powders. The role of shear strain in the refinement, synthesis, and consolidation of the composite was investigated. The comparison between HPT and BM was carried out and compared to other methods in order to find the best approach to fabricate MMCs in the future.

2. Experimental Procedure

The starting materials used in this research work were copper (Cu), niobium (Nb), and graphite (C) powders with different compositions corresponding to Cu-1, 5, and 15 vol%NbC in order to study the effects of NbC volume fraction on the mechanical and electrical properties and the formation of the Cu-NbC composite. Cu powder was of 99.9 wt% purity with particle size ≤180 μm (Wako-Japan); Nb powder was of 99.5 wt% purity with particle size ≤7 μm (Wako-Japan); and C powder was of 99.9 wt% purity with particle size ≤10 μm (Wako-Japan).

The HPT process was conducted at room temperature with equipment as shown in Figure 1. The equipment consists of upper and lower anvils having a shallow circular hole of 20 mm in diameter and 0.25 mm depth at the center. A mixture of Cu-Nb-C powders was placed in the hole and compacted at pressures of 1.25 and 5 GPa. Subsequently, the lower anvil was rotated relatively to the upper anvil under the imposed pressure of 1.25 or 5 GPa with a rotation speed of 0.5 rpm. The Cu-15 vol%NbC premixed powder was HPTed under a pressure of 1.25 GPa and 10 turns for testing the ability of refinement, formation, and consolidation of the composite powder. The Cu-1 and 5 vol%NbC premixed powders were HPTed under a pressure of 5 GPa and 20 turns for investigation of electrical and mechanical properties. No process control agents were used during HPT.

In the BM method, a mixture of Cu-Nb-C powders was milled using a Fritsch "Pulverisette 5" planetary ball mill under argon atmosphere at a rotation speed of 250 rpm for 30 h. The balls used were 10 mm in diameter and powder-to-ball weight ratio was fixed at 1 : 10. 1 wt% of ethanol was used as a process control agent in order to prevent excessive cold welding of these powders during BM. The SUJ-2 balls (chrome steel ball) which have high toughness and wear resistance were chosen in order to reduce contamination during ball collisions. In order to prevent oxidation of the powders, loading and discharging of the powders were carried out inside an argon-filled glove box.
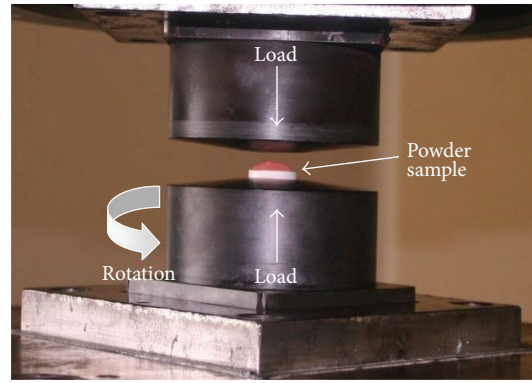

FIGURE 1: Picture of HPT apparatus and processing.

The formation of NbC in the copper matrix after HPT and BM (labelled as-HPTed and as-milled) was identified using an X-ray diffractometer (XRD; RIGAKU RINT-2500 X-ray diffractometer) with CuKα radiation. Microstructures of the as-HPTed Cu-15 vol%NbC sample were investigated using a scanning electron microscope (SEM; LEO Supra 55 VP). The as-HPTed Cu-1 and 5 vol%NbC samples were annealed at 700°C for 1 h in vacuum. The as-milled Cu-1, 5, and 15 vol%NbC powders were consolidated by spark plasma sintering (SPS) using a Dr. Sinter @ 2040 spark plasma sintering system (Sumitomo Coal Mining, Tokyo, Japan) at 1000°C, 100 MPa for 10 min. These sintered temperature and pressure were chosen in order to optimize between density, electrical conductivity, and mechanical properties.

The microhardness and electrical conductivity were measured using a Vicker's microhardness tester and Ulvac ZEM 1 electrical conductivity equipment, respectively. Electrical conductivity was measured using the IACS unit whereby IACS refers to the International Annealed Copper Standard and 100% IACS is equal to 58.0 m/Ωmm^2. The density of the as-HPTed and SPS Cu-NbC milled samples was measured using Archimedes' method. It should be noted that, for HPT samples, tests were conducted at the region $r \geqq 3.5$ mm where a homogeneous microstructure was observed. Three tests were conducted for each measurement. An error of approximately ±3 in the microhardness test and an error of 1% in both the density and electrical measurements were observed.

3. Results and Discussion

3.1. The Formation and Refinement of In Situ Cu-NbC Composite Powder. Using SEM micrographs and microhardness measurement along the diameter of the as-HPTed Cu-15 vol%NbC sample, a schematic illustration of microstructure evolution of Cu-Nb-C powders after HPT is given in Figure 2(a). In Figure 2(b), at the center region of the sample where the shear strain was virtually zero and it had undergone nearly pure compression [31], original Nb particles were unable to refine and high porosity was observed. In the region $r \sim 1.5$ mm, most Nb particles were plastically deformed resulting in the formation of a layered structure. In the region
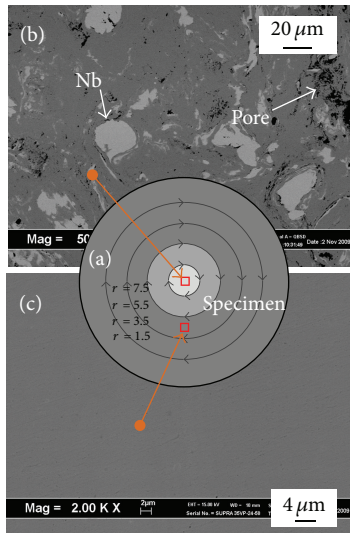
FIGURE 2: (a) Schematic illustration of microstructural evolution under shear strain and SEM micrographs of the as-HPTed Cu-15 vol%NbC at (b) 0 mm and (c) 3.5 mm from the center of the sample.
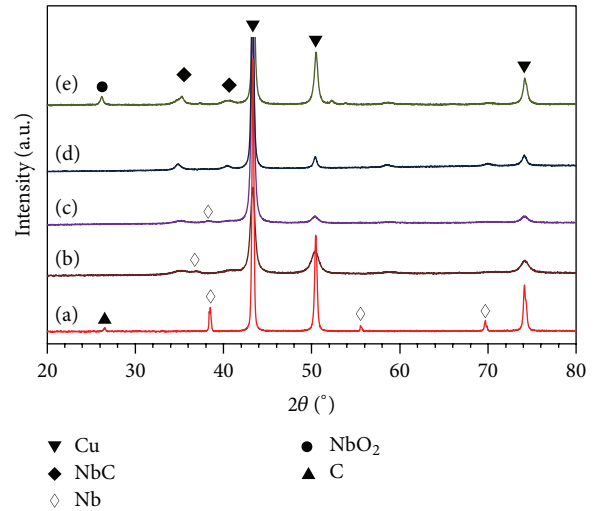

FIGURE 3: XRD patterns of (a) mixture of Cu-Nb-C powder, (b) the as-milled Cu-15 vol%NbC powder after 30 h milling, (c) the as-HPTed Cu-15 vol%NbC compact after 20 min of HPT deformation, (d) the as-HPTed Cu-15 vol%NbC after annealing, and (e) the as-milled Cu-15 vol%NbC compact after SPS.

$r \sim 2.5$ mm, thin Nb layers were fractured into small particles and homogeneously distributed in the copper matrix. In the region $r \sim 3.5$ mm, Nb particles were substantially fractured into very fine particles and homogeneously distributed in the copper matrix. In the region $r \geqq 3.5$ mm, where it had undergone large shear strain ($\gamma \geqq 250$), full density was obtained with extremely fine Nb distribution in the copper matrix which was undetectable by SEM even at high magnification (Figure 2(c)). This indicates that the large shear strain is an important factor to refine powder particles. The detail of microstructure evolution was described in a previous research [33]. In previous research work [14], it was shown that nanoscale NbC was formed *in situ* within the Cu matrix after ball milling for 30 h.

The *in situ* formation of NbC in the copper matrix by HPT and BM was investigated using XRD, as shown in Figure 3. Figure 3(a) is the XRD pattern of a Cu-Nb-C powder mixture. The peaks of Cu, Nb, and C can be clearly observed. However, after BM and HPT, the peaks of Cu, Nb, and C become broadened (Figures 3(b) and 3(c)). This is due to high strain and fine particle size. It can also be seen that the XRD patterns of Cu-Nb-C powders after BM and HPT are quite similar. In BM, NbC was formed after 30 h milling. Similarly, the formation of NbC was reported to be only after 20 h of milling [34]. This difference in the duration of NbC formation by BM may be attributed to different milling conditions. However, NbC was formed after only 20 min of HPT deformation (10 turns). This indicates that, similar to BM, the mechanical alloying process also took place during HPT but within a shorter duration. This is probably due to the fact that HPT can impose a certain high level of strain in a shorter duration with sufficient activation energy to accelerate the reaction between Nb and C. The mechanism of the formation of NbC

by mechanical alloying had been discussed in more detail in a previous research work [35].

After annealing, the peaks of NbC become more evident and no oxidation can be observed in the HPTed sample (Figure 3(d)). However, in the as-milled Cu-NbC sample, NbO_2 was formed after SPS (Figure 3(e)). This is due to the reaction between Nb and O_2 from the process control agent used. Besides, the EDX analysis results also showed that Fe was introduced into the composite sample from the grinding media during ball milling [35].

3.2. Consolidation of In Situ Cu-NbC Composite Powder.

Two processes simultaneously took place during HPT, that is, compaction and torsion. After HPT, the full density of as-HPTed Cu-NbC composites was obtained without any need of subsequent sintering process, as shown in Figure 4. The maximum density of the milled Cu-NbC composites obtained was only 81 and 98% of the theoretical density after using normal sintering [14] and SPS (Figure 4), respectively. The density of other composite materials is also presented in Figure 4; that is, the density of Fe-TiC [17], Al-Al_2O_3 [5], and Cu-Ta [18] composites was reduced with increasing volumes of reinforcement. In these research works, Fe-TiC and Cu-Ta composites were fabricated by milling the matrix (Fe, Cu) and reinforcement (TiC, Ta) powders together and subsequently compacted and sintered. The Al-Al_2O_3 composite was formed from Al and Al_2O_3 powders using ECAP at 200°C. The low density of these composites was due to the difficulty in the deformation of hard reinforcement particles in the matrix during consolidation [8, 9]. It was reported that the yield strength of the composite powder increased with increasing volume fraction of reinforcements [9, 36]. The increase of yield strength led to a restriction in the
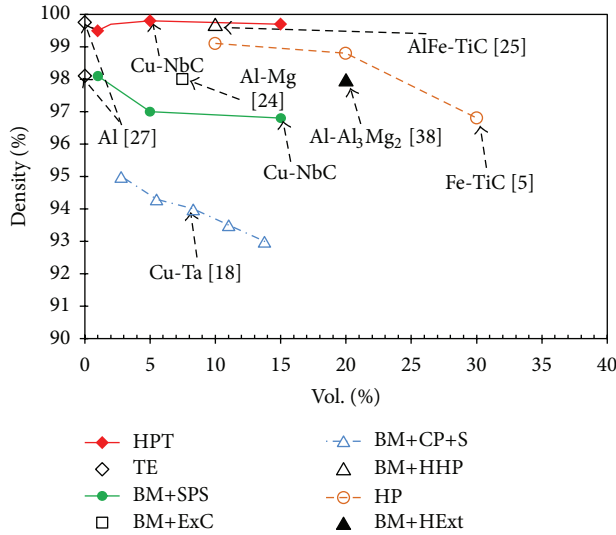
FIGURE 4: Density of different composites and alloys fabricated by different techniques and consolidation methods. HPT: high pressure torsion, BM+SPS: ball milling followed by spark plasma sintering, BM+CP+S: ball milling followed by cold compaction and sintering, HP: hot pressing, TE: torsion extrusion, BM+ExC: ball milling followed by explosive consolidation, BM+HHP: ball milling followed by high pressure hot pressing, and BM+HExt: ball milling followed by hot extrusion.

deformation of particles to fill in the pores and, subsequently, inhibited the densification process [17].

Using HPT, composite powders were severely deformed by large shear strain under high compaction pressure and the pores were easily removed even at high volume fractions of reinforcement. The role of shear strain in cold consolidation can be assessed by the density of Cu-15 vol%NbC compact before and after HPT. As a result, the green density of the compact under pressure of 1.25 GPa without torsion was only 91.0% of the theoretical density. Another research work also reported that compaction of powders was not feasible by mere application of a pressure, even at 6 GPa, without rotation to induce strain [31]. This indicates that large shear strain plays a key role to consolidate composite powders to full density. Currently, the role of shear strain in the consolidation of powders was also well recognized by other research works [5, 27, 37].

With this successful refinement, consolidation, and synthesis of full density MMCs, HPT promises to be a potential low-cost method for the fabrication of MMC powders, alloys, and intermetallics in the future. Besides, HPT had also been reported to enhance the consolidation process of ceramic materials even at low sintering temperatures [31]. Furthermore, HPT shows to be superior in the consolidation of composite powders than equal channel angular extrusion (ECAE) [37], hot extrusion [38], TE [27], and ECAP [5] where heat (400, 400, 350, and 200°C, resp.) was needed during consolidation.

3.3. Properties of In Situ Cu-NbC Composite Fabricated by HPT and BM. Electrical conductivity and microhardness of
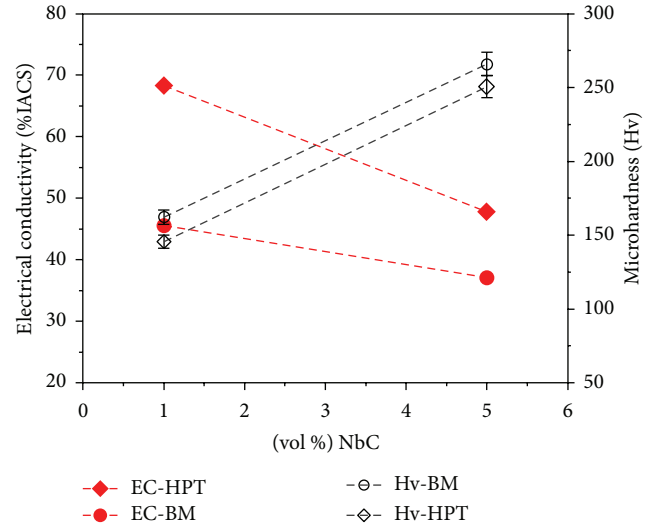

FIGURE 5: Comparison of electrical conductivity of Cu-NbC composites fabricated by HPT and BM methods; EC-HPT and Hv-HPT are electrical conductivity and microhardness of HPTed Cu-NbC composites, respectively; EC-BM and EC-BM are electrical conductivity and microhardness of milled Cu-NbC composites, respectively.

HPTed and milled Cu-NbC composites are plotted together in Figure 5. It can be seen that, using HPT as a fabrication method, the electrical conductivity of the Cu-NbC composites is significantly increased compared to composites synthesized using BM and sintered by SPS. For instance, at 1 vol%NbC, the electrical conductivity of the HPTed Cu-NbC composites is 50% higher than that of the milled Cu-NbC composite, whilst the microhardness of HPTed Cu-NbC composite is only 9% lower than that of the milled Cu-NbC composite. The large improvement of electrical conductivity of the HPTed Cu-NbC composites is considered to be attributable to its higher density and virtually nonexistent contamination. The higher microhardness observed in the milled Cu-NbC composite is possibly due to the introduction of Fe and oxides in the copper matrix during milling and sintering [35].

4. Conclusions

HPT is a self-cold consolidation process where composite powders can be consolidated to full density at room temperature. The large shear strain of HPT plays the most important role in the refinement, synthesis, and consolidation of fully densified Cu-NbC composite. Even though both BM and HPT can synthesize *in situ* Cu-NbC composite, HPT can perform the process in a much shorter duration (20 min).

The HPTed Cu-1 vol%NbC composite fabricated using HPT showed high electrical conductivity in combination with high microhardness (68% IACS and 150 Hv) and high thermal stability (700°C). The great improvement of the electrical conductivity of the HPTed Cu-NbC composite compared to that of milled Cu-NbC composite was due to

its higher density and nonexistence of contamination. HPT is not only a powerful tool for the synthesis of *in situ* Cu-based composite powders but also a promising technique for the synthesis of other metal matrix composites.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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