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Plastic deformation and microstructural evolution during the shock consolidation of ultrafine copper powders

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ABSTRACT

Shock consolidation of ultrafine copper powders at room temperature for bulk nano/ultrafine structured materials is achieved in a gas gun system. The stress states in the powders during the shock consolidation process are analyzed using the finite element method associated with the dynamic densification model (P-α model). The mechanical properties of the shock-consolidated copper are evaluated in terms of hardness, static tensile and compressive strengths, and dynamic compressive strength. The microstructures are characterized using an optical microscope, a scanning electron microscope, and X-ray diffraction (XRD). The XRD patterns are quantitatively analyzed in order to estimate the crystallite sizes and dislocation densities using the Convolution Multiple Whole Profile method. The shock-consolidated specimens were highly densified over 98% of relative density with uniform spatial distributions of high hardness. However, insufficient consolidation due to the tensile stress wave induced by the interactions between shock waves in the powders and due to the ultrafine particles requiring high pressure for good bonding has resulted in several defects in the consolidated specimens. These defects cause tension–compression asymmetry in the shock-consolidated materials. Compared with the tensile results, where fractures occurred at low stresses without plastic deformation due to weak interparticle bonding, the high compressive yield stresses of 600 and 900 MPa with large plastic strains are achieved in the static and dynamic compression results, respectively. These high compressive flow stresses are attributed to the extremely high dislocation density and the refinement of the crystallite size via the shock deformations. A microstructure model is proposed for the extremely high dislocation density, where dislocations are generated not only by shock waves but also by plastic flow during the void collapses. The strengths of the shock-consolidated specimen are slightly decreased during the post-shock deformations due to decreases in the excess dislocations despite further refinement of the crystallite size.

1. Introduction

Microstructures and chemical compositions are the primary factors that determine the mechanical properties of metallic materials: for example, the grain size and dislocation density are well-known parameters for controlling material strengths. When a polycrystalline material has a sub-μm grain size, which is smaller than conventional grain sizes, the material is defined as a nano or ultrafine grained (UFG) material. In general, the conventional grain size is in the range of 1 mm to 10 μm, UFG materials are from 1 μm to 100 nm, and nanocrystalline materials are < 100 nm [1]. Nano/UFG materials have significant potential in industrial applications due to their higher strength/hardness, higher toughness [2,3], higher diffusivity [4–6], higher specific heat [7], and enhanced thermal expansion coefficient [8] compared with their coarse grained polycrystalline counterparts [9]. In particular, the plastic strengths of the materials increase with decreases in grain size, following the Hall–Petch relation [10,11]:

$$\sigma = \sigma_o + kd^{-1/2}, \quad (1)$$

where σ is the strength, σ_o is the lattice friction stress required to move individual dislocations in coarse-grained reference materials, d is the grain size, and k is a positive material constant referred to as the Hall–Petch slope.

These excellent properties and potentials of nano/UFG materials are the driving force of active research for practical

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applications, and they provide scientific meaning in understanding the underlying physics of grain refinement in materials. As a result, the interest and effort of many researchers in materials science and engineering have been focused on the microstructure and mechanical properties of nano/UFG materials.

There are two primary approaches for the fabrication of 'bulk' nano/UFG materials: top-down and bottom-up approaches. Top-down approaches are where nano/UFG structures are produced through machining or deforming the initial bulk material with coarse grain sizes. The deforming grain refinement technique is generally represented as severe plastic deformation (SPD) [12]. There are several commonly used SPD processes: equal-channel angular pressing (ECAP) [13–15], accumulative roll-bonding (ARB) [16], high-pressure torsion (HPT) [17–19], and so on. The nano/UFG microstructures produced via the SPD processes include UFG microstructures and high densities of crystalline defects, such as vacancies, dislocations, twins, and grain boundaries. Bottom-up approaches are methods where nano or sub-μm particles are used as the starting materials for synthesizing bulk nano/UFG materials via powder metallurgy. Well-known bottom-up processes are inert gas condensation [20], electro-deposition [21,22], and ball milling that produces nano grained powders and subsequent conventional consolidation [23,24], spark plasma sintering [25–27], high pressure cold sintering [28], and shock wave consolidation [29,30] techniques. In the bottom-up methods, it is important to maintain and control the microstructures of the starting nano/UFG materials during the consolidation process. If these requirements are satisfied, then bottom-up methods are more effective in controlling the properties of the final bulk material than top-down methods.

Shock wave consolidation (in short, shock consolidation) is a bottom-up approach process, where powders are densified and consolidated using shock waves with high pressures (several or tens of GPa) during very short times (several μsec). During the shock consolidation process, the powders and powder agglomerates are collapsed through compressing them at high pressure for short times and generating dynamic flows on the surface of the powders using the movement of the high velocity powders. These pressure- and rate-intensive interactions between the surfaces of the particles can enhance powder–powder bonding in order to achieve bulk shapes.

Many types of metallic powders, including particles with nano-size or nano-scale microstructures, have been consolidated using shock waves [31–45]. For example, Zhang et al. [42] consolidated 30–50 nm silver nano-powders using shock waves for fully dense bulk nanostructured components; Nieh et al. [31] also produced shock-compacted nanostructured bulk from aluminum powders with a size of 40–50 nm. These results have demonstrated shock-consolidated bulk materials with high densities. Although there have been many results that exhibit high relative density after shock consolidation in previous works, the microstructural features, such as defects and shapes, have predominantly been considered qualitatively with few quantitative analyses. More importantly, sufficient investigation of the mechanical properties of the final bulk parts processed via shock consolidation in conjunction with the microstructure features has rarely been investigated.

When a bulk material is deformed by shock waves, more internal defects, e.g. vacancies, dislocations, and twins, or metastable microstructures are generated than those deformed by conventional static loading [46]. These defect effects also operate during the shock consolidation and are influenced by the features of the shock waves, particle geometry, and material behaviors during the shock consolidation process. For example, Gourdin [32] applied the shock consolidation process to coarse spherical copper powders and observed stream type microstructures, which are not usually observable morphologies in conventional copper. That is, the microstructure of the consolidated bulk sample depends significantly on the shock wave characteristics. Although there have been numerous shock consolidation results in previous works, these microstructural features, i.e. defects and shapes, have predominantly been considered qualitatively with few quantitative analyses. More importantly, investigations of the mechanical properties of the final bulk parts processed using shock consolidation have rarely been conducted. Because the microstructure critically affects the physical properties of the consolidated bulk sample, the quantitative analysis of the microstructure and defects in accordance with the effect of the shock waves should be included when applying shock consolidation, which results in unusual microstructures.

In this study, the shock consolidation process was applied to the sub-μm copper powders in order to obtain a nano/UFG bulk. The hardness measured in previous research was investigated as well as the static and dynamic mechanical properties. The qualitative and quantitative experimental analyses of the evolutions of the microstructure and defects during the shock consolidation and post-shock deformation were performed concurrently; these microstructural and mechanical results are analyzed in relation to the deformation behaviors using finite element method (FEM) simulations.

2. Experimental procedure

Commercially available copper nano-powders with $>99.9\%$ purity and an average particle size of 100 nm produced using a pulsed wire evaporation method [47] were used. Although the powders were handled carefully and kept in a vacuum, oxide layers on the nano-particle surfaces cannot be fully prevented. In order to remove these oxide layers, a hydrogen reduction process was implemented with heating and cooling rates of 3 °C/min at 350 °C, respectively, and they were maintained in a hydrogen gas atmospheric condition for 60 min.

Fig. 1 presents the scanning electron microscopic (SEM) images of the powders before (a, b) and after (c, d) the hydrogen reduction process. The initial powders were naturally agglomerated. The hydrogen reduction-processed copper powders grew and combined to have an average particle size of approximately 400 nm. The initial nano-powders and hydrogen-reduced powders are referred to as "NP" and "HRP", respectively.

In order to perform the shock consolidation process, a single gas gun system was designed and installed. In this system, the propellant gas can be compressed up to 2000 psi; furthermore, a projectile with a 40.0 mm diameter, which was accelerated by the compressed gas, impacted a target through a 3 m barrel. The schematics of the projectile and target are plotted in Fig. 2. The projectile for the impact part was composed of an exchangeable steel head and a polycarbonate sabot. The accelerated projectile impacted the powders that were inserted in the target. The target system was positioned in a vacuum tank at $\sim 2.0 \times 10^{-1}$ Torr, which was designed for the safe collections of the shock-consolidated samples. The target was made from mild steel; a momentum trap with the same material was attached to the back of the target in order to minimize the effect of shock reflections on the free surface. Shrink fitting was applied during the fabrication process of the target in order to improve the quality of the interfaces between the parts. A powder room (diameter = 10.0 mm and height = 5.0 mm) was placed 5.0 mm behind the impact surface. The HRPs were packed in the powder room with an initial relative density of approximately 95%. In order to generate a planar shock wave, which is required for uniform shock consolidation, the projectile was aligned with the target using a laser calibrator. In the study, nitrogen gas was used as the propellant