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Technical Paper

In-situ method to produce nanograined metallic powders/flakes via ultrasonic shot peening

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ABSTRACT

In this study, nanograined metallic powders/flakes were successfully produced by severely ultrasonic shot peening for the first time according to the author's knowledge. Surface nanocrystallization of the material was realized and then the fabricated nanograined layer was impacted in-situ by the subsequently ultrasonic shot peening. The repeatedly impacts on the nanograined surface layer result in the fracture of the materials and the formation of the metallic powders/flakes due to the significant drop of the ductility and work-hardening of the nanograined surface. Transmission Electron Microscope (TEM) observations indicate that the generated metallic powders/flakes are consisting of the nano grains with the size in the range from 20 nm to 100 nm and the nano lamellar with the average thickness of 50 nm. Micro-crack initiation and propagation were also characterized at the topmost nanograined surface layer. Research results suggested that the mechanism for the formation of the nanograined metallic powders/flakes during the ultrasonic shot peening includes the stages of surface nanocrystallization and fracture of the fabricated nanograined surface and ultrasonic shot peening can be potentially used as an effective method to produce nanograined metallic powders/flakes.

1. Introduction

Nanograined (NG) materials, with average and range of grain sizes typically smaller than 100 nm, have attracted more and more attentions from the materials community for decades [1–7]. Contrary to conventional coarse-grained counterparts, NG materials exhibit peculiar and interesting mechanical, physical and chemical properties, e.g. increased mechanical strength, enhanced diffusivity and higher specific heat [8–11]. Due to these peculiar and interesting properties, NG materials are experiencing a rapid development in recent years for their existing and/or potential applications in a wide variety of technological areas such as electronics, catalysis, batteries, ceramics, magnetic data storage, structural components and so on [12–16].

Conventional coarse-grained metallic powders/flakes have been widely used in the surface coating technology and polymer composites. It has been proved that the metallic powders/flake could improve the wear resistance [17], corrosion resistance [18,19] and scratch resistance [20] of the coatings. The metallic powders/flake could also be utilized as the conductive filler to produce polymer composites used as shields against electromagnetic interference [21] and electrically conducting thermoplastic composites [22]. Due to the significant increase in hardness, strength and electrical conductivity of NC metals and alloys [23–26], the metallic powders/flake with nanocrystalline structure promise a potential engineering/scientific application in the field of surface coating and polymer composites et al.

Methods for generating NG metals and alloys generally include severe plastic deformation (SPD) [27], mechanical alloying [28], electrodeposition [29] and sputtering [30] et al. As one of the SPD methods, ultrasonic shot peening (USP) has the advantage of high efficiency and has been successfully used in forming nanograined materials at the surface of the metallic workpiece [31–34]. Several published papers indicate that nanograined surface layer could be successfully generated via USP in pure iron [35], copper [36] and other metals and alloys [37–39]. Currently, methods capable of producing metallic powders/flakes consisting of polycrystalline nanograins include ball milling [28] and rapid solidification of small liquid droplets followed by annealing/heat treatment [40]. Large metallic powders are ball milled for many hours or even days to create nanograins in the powders. This method, however, suffers

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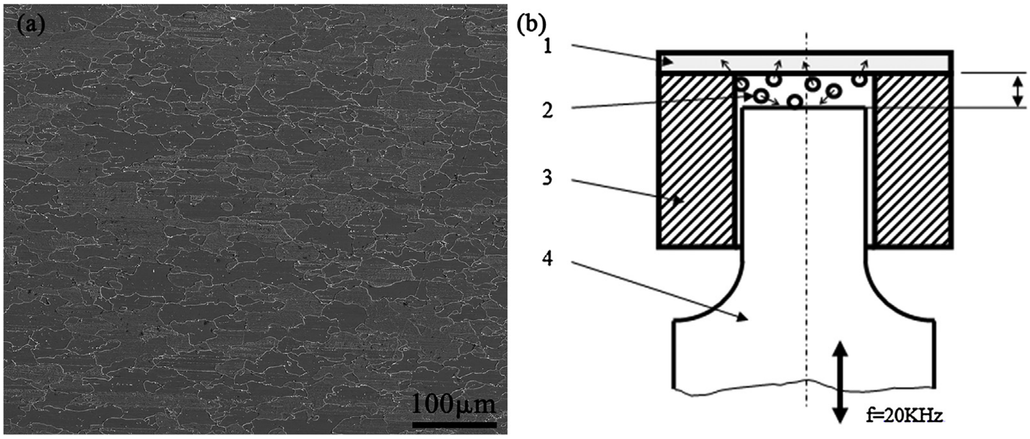

Fig. 1. (a) Microstructure of the as-received AISI-1018 steel with coarse grains and (b) Principle of ultrasonic shot peening. The setup consists of (1) metal sample, (2) shots, (3) enclosure and (4) ultrasonic horn.

from contamination from the interactions between the powders and the balls or the internal walls of the container.

In this study, an in-situ method to produce nanograined metallic powders/flakes via ultrasonic shot peening was firstly proposed and released. The surface layer of the bulk solid sample, which is made from AISI-1018 steel, was severely plastic deformed by using repeatedly striking of shots driven by high intensity ultrasonic vibration. Surface nanocrystallization was realized at the beginning of the severely ultrasonic shot peening. The severe plastic deformation induced nanograined surface layer can result in a record-high strength, however the ductility and work-hardening are decreased considerably [41]. An example from Chen et al. [42] shows that a nanograined 316L stainless steel has a yield strength as high as 1450 MPa, but its work-hardening diminishes and elongation-to-failure drops to only ~3%. The significant drop of the work-hardening of the generated nanograined layer makes the materials at the topmost surface layer very brittle. By the continuous striking of the high-speed balls, cracks initiated and propagated in the nanograined surface layer, resulting in the formation of the nanograined metallic flakes. The fractures are freshly formed in-situ during severely ultrasonic shot peening and the fracture surface is not in contact with a liquid or with solid balls. Hence, the nanograined metallic powders/flakes fabricated in this method are much cleaner than those produced using ball milling or using rapid solidification methods.

2. Materials and methods

2.1. Materials and ultrasonic shot peening

The AISI-1018 steel plate with the thickness of 3 mm was used for severely ultrasonic shot peening. Fig. 1(a) shows the microstructure of the as-received materials. The grain size is in the range from 50 μm to 200 μm. Fig. 1(b) shows the principle of the ultrasonic shot peening. Steel shots with the diameter of 5 mm were placed on the surface of the ultrasonic horn in this case. The ultrasonic horn was connected to a transducer and generator of ultrasonic signals of 20 kHz. Driven by the ultrasonic signal, the surface of the ultrasonic horn will be vibrated. The surface layer of the bulk solid sample, which is made from 1018 steel, was severely plastic deformed by using repeatedly striking of shots driven by the high intensity ultrasonic vibration. The experimental setup designed in this study is similar with the experimental setup in the authors' previous studies [43,44].

2.2. Materials characterization

The morphology of the fabricated nanograined metallic flakes was observed by the Leica optical microscope. Scanning electron microscope (SEM) observations were performed on a FEI QUANTA-3D FEG scanning electron microscope. The cross-sectional SEM specimen was first mechanically polished using diamond paste, and then etched at room temperature in a solution of 100 mL alcohol and 4 mL nitric acid. The characterization of the finer details of the microstructure in the generated metal flakes was performed using an FEI Tecnai G20 transmission electron microscope equipped with the LaB6 filament and operated at 200 kV. The specimens for TEM examination were prepared by the FIB lift-Out method using FIB/SEM Dual Beam FEI Nova 200 [45]. The bright-field (BF) TEM images as well as select pattern diffraction were taken to characterize the microstructure of the materials.

3. Results

Fig. 2(a) shows the nanograined metallic flakes fabricated by ultrasonic shot peening. The morphology of the metallic flakes was observed via optical microscope. The size of the generated metallic flakes is in the range from tens of micrometers to hundreds of micrometers as shown in Fig. 2(b) and (c). The generated metallic flakes are very clean and shiny, there are no oil and other contaminants on the surface of the metallic flakes due to the in-situ severely ultrasonic shot peening method.

The TEM sample was cut from the metallic flakes by Focus Ion Beam (FIB) and lifted out via the micromanipulator equipped with Omni probe as shown in Fig. 3(a). The sample was thinner to 100 nm thickness by ion beam subsequently for TEM characterization. Fig. 3(b) is the bright field TEM characterization of the microstructure of the metallic flakes. The nano grains with size in the range from 20 nm to 50 nm were characterized between the lamellar-shaped nano grains. The lamellar-shaped nano grains with the thickness of or less than 50 nm are orientated. Insert of Fig. 3(b) shows the selected area diffraction pattern, which indicates that the generated metallic flakes are consisting of randomly orientated nano-sized grains.

As indicated by red dash lines in Fig. 3(b), the majority of the generated nanocrystalline grain is of lamellar-shaped with the almost the same orientation. And there are some smaller equiaxed grains indicated as the blue dash lines in Fig. 3(b). High density of the grain boundary was obtained in the metallic flakes. The high density of grain boundary is good for the improvement of the mechanical properties of materials because grain boundaries will terminate the