Synthesis of the SUS 316L Powders with a Nano-Meso Bi-Modal Structure

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Abstract: Pure stainless steel (SUS 316L) powders with both a nano-grain and micro-structure were prepared using a mechanical milling process. The bimodal microstructure consists of a nano-grain with a size of 50 nm in the surface region and microstructure in the core of the particle. The nano-grain with a bcc structure at the surface and the microstructure of fcc in the core of the particles were prepared by milling at 150rpm for 9h, and those compositions were 14.39% for the nano structure and 85.61% for the micro structure, respectively. Spark plasma sintering (SPS) was carried out for the compaction of the powders. The compacts forming powders with a bi-modal structure have a mesoscopic structure.

Keywords: SUS316L, mechanical milling, spark plasma sintering, hybrid microstructure.

1. INTRODUCTION

Recently, many researchers have been interested in high strength materials with nano grains. The nano-grain refinement using a severe plastic deformation process is very effective at achieving high strength properties of the metallic materials [1-2]. In particular, the microstructure design was focused upon because of the enhancement of the mechanical, physical, and chemical properties of the material. The powder metallurgy process (PM) is a new process combining mechanical milling (MM) or mechanical alloying (MA), heat treatment, and sintering [3-5]. Significantly large deformations during the milling treatment lead to a powder with a nano grain structure. These powders with nano grains by milling show not only high strength but also low ductility [3]. Regrettably, the poor ductility of uniform nanocrystalline affects the plastic instability. Fujiwara *et al*. reported a heterogeneous micro-structure with both nano- and micro- grains attributed to a high strength and sufficient plastic strain, simul-taneously [6-7]. Although the bimodal microstructure is difficult to obtain by a conventional mechanical processing, it is easily achieved by the MM and subsequent sintering processes, such as hot roll sintering (HRS) and spark plasma sintering (SPS) process [4-9]. A high density sintered compact can be made within a short time using the SPS method. SPS was proposed as a novel sintering method for a sintered compact with high density for a short period of time [10-11].

In this study, the progress of the forming nano-grains during MM process was investigated to get an informative formation mechanism for nanomeso struc-ture. Compacts of the powders with a heterogeneous microstructure were carried out using spark plasma sintering (SPS). The heterogeneous microstructure was performed during compaction as well as milling. The concentration of the heterostructure was measured using Mössbauer spectroscopy.

2. EXPERIMENTAL PROCEDURE

The MM treatment of SUS316L (C: 0.024, Si: 0.83, Mn: 0.74, P: 0.023, S: 0.002, Ni: 12.40, Cr: 7.38, Mo: 2.11, Fe: bal. (mass%)) stainless steel powder was performed by horizontal ball mill equipment with a water cooling system in an Ar atmosphere of 2 bar without any process control agent. The MM equipment is a planetary mill (P-100, Teamyung Science Co.) with a SUS 304 stainless steel vial and balls. The MM process was carried out at 400rpm and 150rpm for 3, 6, and 9h.

The MM powders were provided for the SPS process. Compaction as well as thermal treatment for the as-dried was performed simultaneously using the SPS at a high pressure of 50 MPa in the temperature range of 1,050°C for 3h. After SPS, the resulting solid products were cooled in an Ar atmosphere. The SPS samples are 20mm in diameter and 3.3mm in height. The compacts were characterized by means of scanning electron microscopy (SEM) and optical microscopy (OM).

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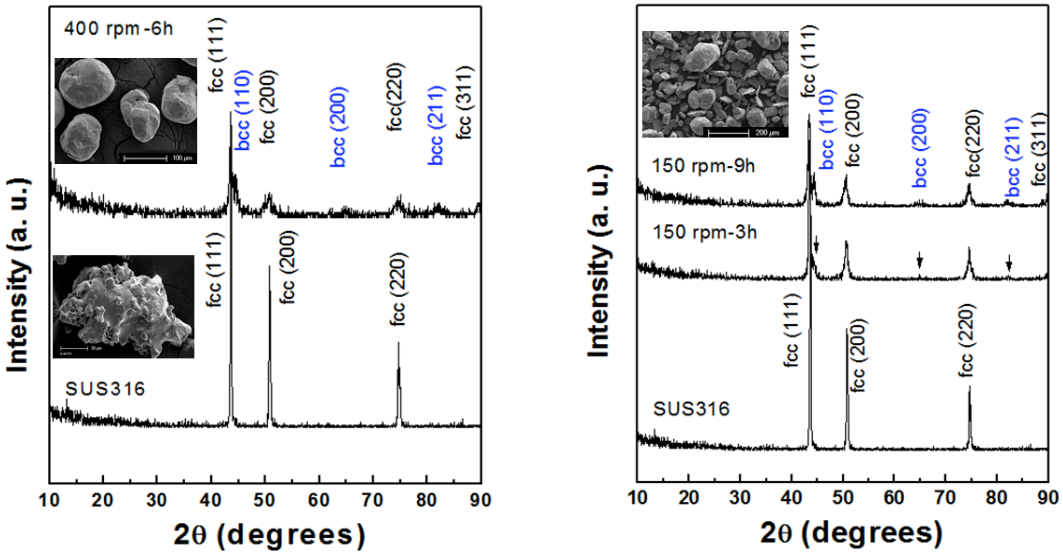
3. RESULTS AND DISCUSSION

SUS316L powders were synthesized by MM treatment using a planetary mill at 400rpm and 150rpm for 3, 6, and 9h. Figure **1**(**a**) shows an XRD pattern for the starting materials and milled SUS 316L powders at 400rpm for 6h. The XRD patterns for the SUS 316L powders milled at 150rpm for 0, 3, and 9h are shown in Figure **1**(**b**). The crystal structure of the starting materials for the SUS 316L is a face centered cubic (fcc). The concentration of the body centered cubic (bcc) phase grows lager, as the milling time of the SUS316L is increased. The milling speed is unrelated with the creation of the bcc phase. Figure **1**(**a**) shows the SEM images for the starting materials of SUS 316L particles and milled particles at 400rpm for 6h. The irregular shape of the starting material was changed into spherical shape particles, after milling at 400rpm. A SEM image of the milled particles at 150rpm for 6h is shown in Figure **1**(**b**). In the results of the XRD and SEM, average particle size of the starting materials was about 500μm. The milled powder at a high speed of 400rpm for 6h shows that the average particle size was about 300μm, after MM treatment. In addition, of the powders was changed from fcc to a bcc structure during the milling.

Cross section images of the powder milled at 400rpm for 6h are shown in Figures **2**(**a**) and **2**(**b**). A relatively homogeneous size distribution of the milled particles was observed. The same grain structures were formed at the core and surface in the milled powder, as shown in Figure **2**(**b**). However, very different grain structures were observed in the milled at



(**a**) (**b**)

Figure 1: XRD patterns for (**a**) the starting materials and milled SUS 316L powders at 400rpm for 6h, (**b**) 150rpm for 0, 3, and 9h. SEM images for the starting materials and milled powders.

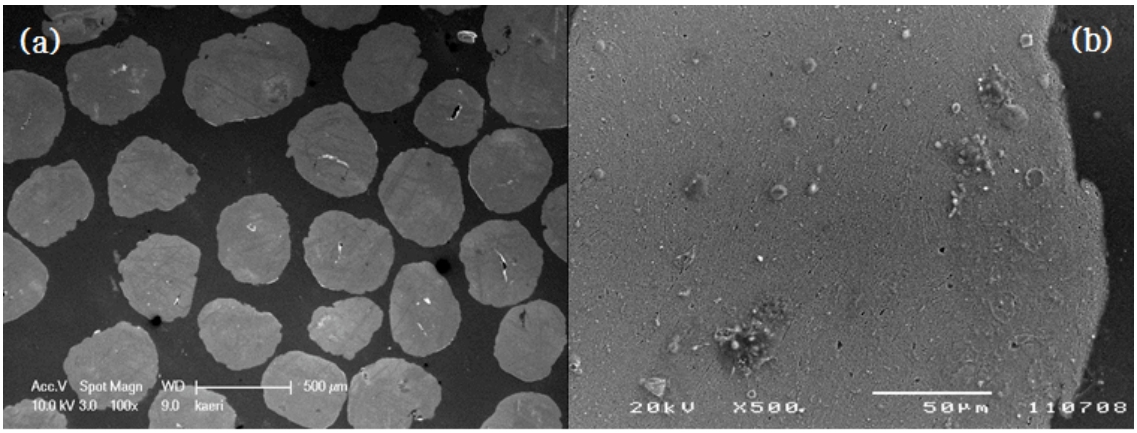


Figure 2: SEM cross section images of the milled powder at 400rpm for 6h. (**b**) Magnification of (**a**).

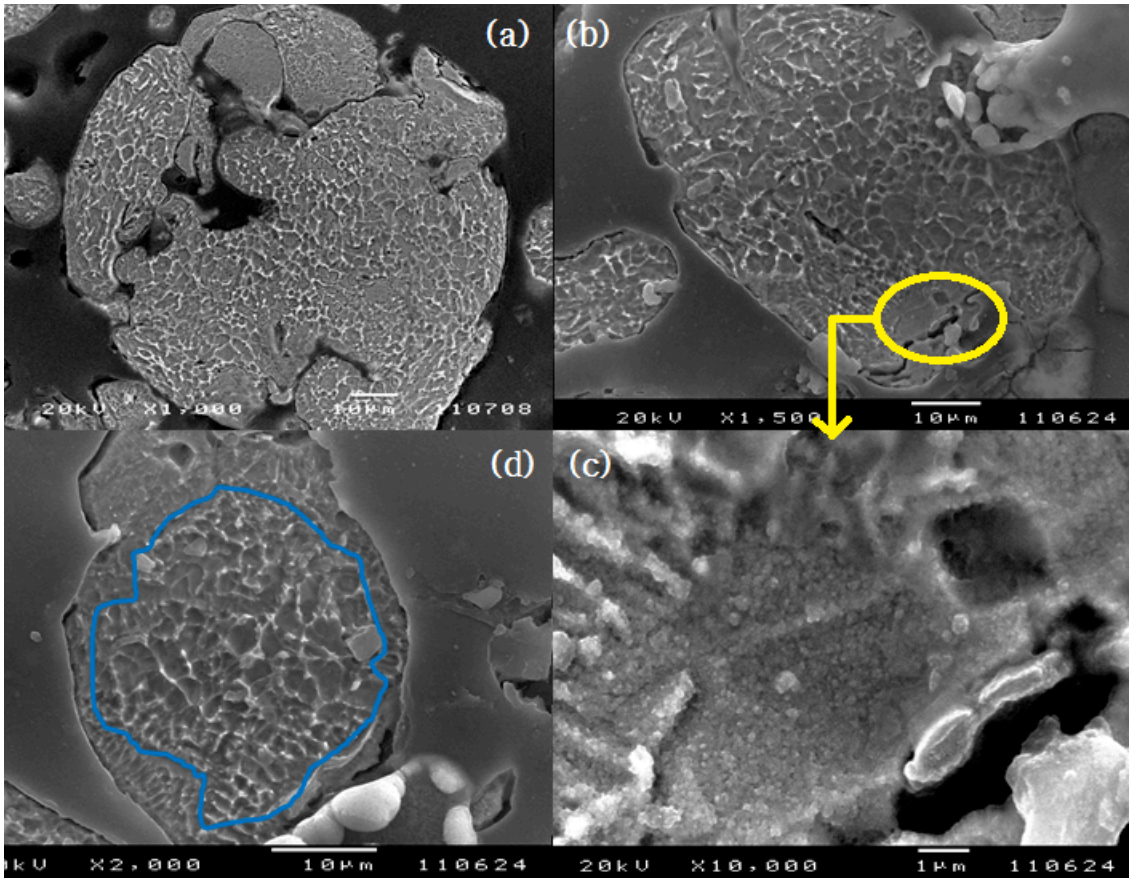


Figure 3: SEM cross section images of the milled powder at 150rpm for (**a**) 3h, (**b**) 6h, (**c**) magnification of circle part of (**b**), and (**d**) 9h.

a low speed of 150rpm. Cross section images of the powder milled at 150rpm for 3, 6, and 9h are shown in Figure **3**. Some of the powders were changed from fcc to a bcc structure during the milling. However, very different grain structures from the core part were formed at the surface layer in the milled powder. Grains with nano sizes at the surface layer were measured. In contrast, micron sized grains were formed at the core in the particles. The results of an XRD and SEM show that, the concentration of both the bcc structure and nano-sized grains were increased, as the milling time was raised from 6 to 9h. This means a bcc structure was formed at the surface layer in the particles. Also, a broad line width was observed in the bcc phase. Nano sized grains were formed in the bcc-phase. These hetero structures with different sizes were crystallized during milling at low speed. This nano-meso hybrid structure is well known to influence the mechanical properties of different materials [1, 12-14]. A part of the fcc phase was deformed during the milling for a very long time, and a phase transition to fcc was carried out. However, the grain growth was controlled owing to the very weak transferred milling energy. Thus, the bcc phase with nano grains was molded. The irregular shape of the particles milled at 150rpm for 3h was formatted, as shown in Figure **3**(**a, b** and **c**). Show a cross section of the milled particles at 150rpm for 6h. The nano grains were observed at the shell layer. The shell layer consisting of nano grains was well shaped as the milling time was increased, as shown in Figure **3**(**d**).

The compositions of the nano-grain with a bcc structure at the surface and the microstructure of fcc in the core of the particles were measured and analyzed using Mössbauer spectra [15-16], as shown in Figure **4**. The Mössbauer spectrum for the SUS 316L was analyzed as one single line as shown in Figure **4**(**a**). In the absorption spectrum of the powder, one can see that the spectrum consists of a single line, where the single line reveals a non-magnetic phase of the SUS 316L. However, the Mössbauer spectrum was analyzed by two single lines for the milled powders, as shown in Figures **4**(**b**) and **4**(**c**). The absorption ratio of the Mössbauer spectrum was increased from 25% to 29%, and to 31%, as the milling time was increased from to 0 to 6h, and to 9h. The compositions of the nano-grain with a bcc structure at the surface and

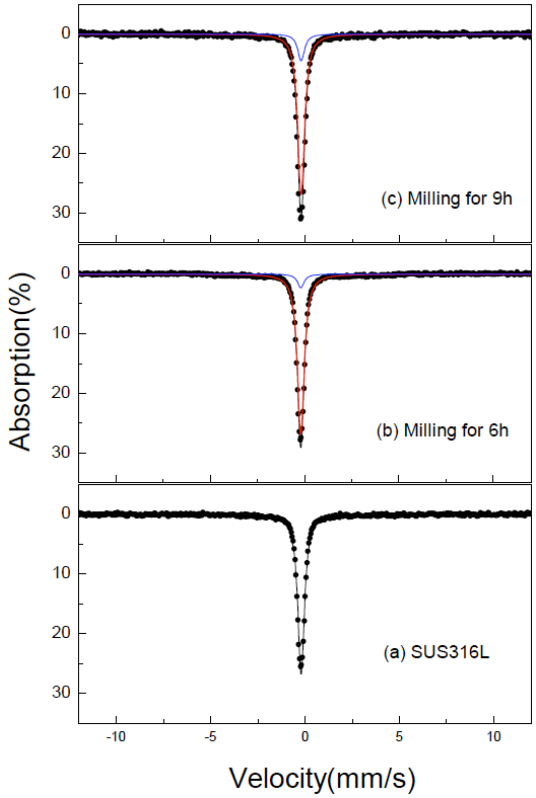


Figure 4: Mössbauer spectra for (**a**) SUS316L, milled powders at 150rpm for (**b**) 6h, and (**c**) 9h.

microstructure of the fcc in the core of the particles milled at 150rpm for 6h were 7.89% and 92.11%, respectively. At previous studies carried out by Ameyama *et al*., the harmonic structures and its mechanical properties of SUS 316L were studied using SEM images and nominal strain-stress curves. The shell with nano grains plays a roll of enhancing the strength, while, the core with micro grains affects to increasing ductility. However, the concentration of the nano-grain in the particles was not measured in the previous studies. In this study, the concentrations of both bcc and fcc structure were analyzed using Mössbauer spectra. The concentration of the nano-grain with the bcc structure was increased to 14.39% for the particles milled at 150rpm for 9h. However, the concentration of the micro-grain with fcc structure was decreased to 85.61% for the particles milled at 150rpm for 9h.

The compaction of milled powders at both high and low speeds was carried out using the SPS method. After the SPS, the cross sections of the compact powder were measured using SEM and OM images, as shown in Figure **5**. The sintered temperature and pressure were 1,050°C and 50 MPa, respectively. The compact particles milled at 450rpm were closed packed. The homogeneous grains and uniform structure of boundaries were well observed, as shown in Figure **5**(**a**). In contrast to Figure **5**(**a**), the compact particles milled at 150rpm for 9h show complicated shapes of the grains and grain boundary. Two areas of bright and dark contrast were observed. The bright area has a smooth surface and the dark area has a rough structure, as shown in Figure **5**(**b**). The bright area with the smooth surface surrounding a dark area corresponds to the core part in milled particles. The dark area results from the shell layer with a nano grain in the milled particles. Ameyama *et al*. reported that this kind of bright area plays a role of enhancing the mechanical strength of the bulk materials. The mechanical properties of compacts with bi-modal will be measured at further studies.

4. CONCLUSION

Mechanical milled powders with a shell and core structure were prepared. When the transferred milling energy resulting from milling speed was weak, the formation of a nano-meso hybrid structure in a particle was achieved. A quantity of nano grains in particles

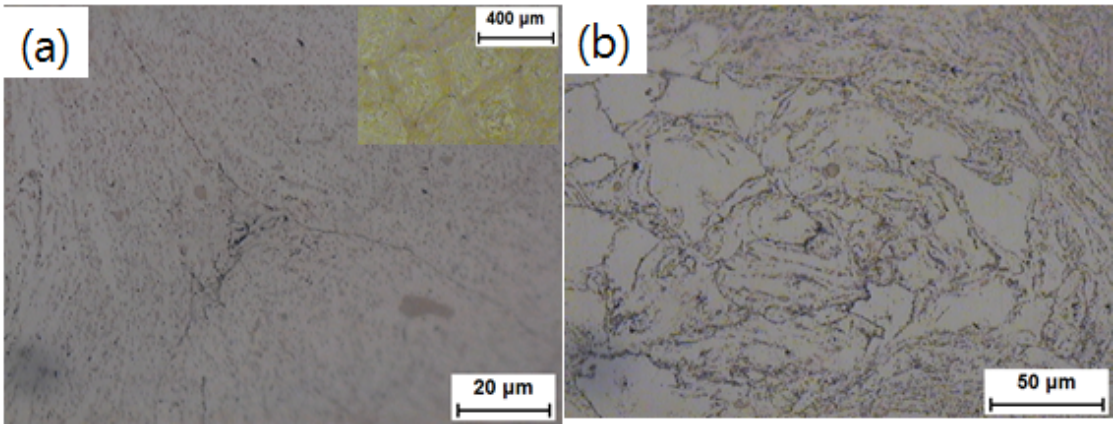


Figure 5: (**a**) SEM and OM images for the compaction of milled powders at 400rpm for 6h, and (**b**) 150rpm for 9h.

was able to be confirmed using Mössbauer spectra, and those concentrations were increased as raised milling time. The shell layer in a particle consists of nano grains. The core part in a particle was formed as micro structure. Nano-meso hybrid structures in a compact prepared by SPS were formed from the particles with bi-modal structure.

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