Enhanced ductility in harmonic structure designed SUS316L produced by high energy ball milling and hot isostatic sintering

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Abstract

The objective of the present study is to optimize the mechanical properties of commercial SUS316L stainless steel through harmonic structure design. The harmonic structured SUS316L compacts were fabricated by a process including high energy ball milling and subsequent hot isostatic pressing. Although the harmonic structured compacts had a bimodal grain size distribution, our investigations showed that both hardness and yield strength still well resembled the Hall-Petch relationship. A great combination of high yield strength and improved uniform elongation can be achieved simultaneously in the harmonic structured SUS316L compacts ($\sigma_y=695\text{MPa}$, $\varepsilon_u=27.1\%$). The improved ductility was attributed to both the strain hardening induced by the coarse-grained structure and the extra strain hardening caused by the peculiar

macroscopic network of ultrafine-grained structure. It was suggested that core grain size of approximately 1.2 μm and shell volume fraction of approximately 50%-60% were the optimized harmonic structure design for austenite stainless steel.

Keywords: Stainless steel; Ultrafine grains; Harmonic structure design; Strength; Mechanical milling;

1. Introduction

Owing to the excellent corrosion resistance, oxidation resistance and good formability, SUS316L stainless steel has been widely used as engineering material in automotive components, high pressure vessels and medical devices [1-5]. However, further application of this material is hindered by its relatively low tensile strength. Grain refinement has been used extensively to produce high strength materials [6-10]. The extremely high yield strength and hardness have been achieved in the ultrafine-grained (UFG) or nanograined (NG) SUS316L stainless steels synthesized by several thermo-mechanical methods, such as surface mechanical attrition treatment (SMAT) [1, 2], equal channel angular pressing (ECAP) [11], high pressure torsion (HPT) [12] and thermal cycling [13-15]. The relationship between grain size and mechanical properties has been investigated in the homogeneous grain-structured SUS316L stainless steels, wherein the yield strength well resembled the Hall-Petch relationship [1, 14]. However, due to the early appearance of plastic instability, ductility tends to be deteriorated in the UFG or NG SUS316L steels [11-15]. Therefore, it is imperative to achieve a good combination of high strength and high ductility in the UFG or NG SUS316L steels for their practical applications.

It is well-known that the bimodal grain size distribution is a simple way in realizing high strength and reasonable ductility. Strength can be influenced by ultrafine grains and ductility can be prompted by coarse grains (CG) simultaneously [16-22]. In recent years, the microstructure design based on multiple scales and multiple layers has been proposed to improve the mechanical properties of metallic materials. “Gradient nanograined structure” has been synthesized successfully in IF steel and Cu, and these metals exhibited extremely large ductility, which was attributed to an extra strain hardening caused by a macroscopic strain gradient [23, 24]. “Trimodal

structure”, consisting of NG or UFG, CG, and ceramic particles, was realized in aluminum based composites. Such structure topology can provide improved mechanical properties and crack propagation resistance [25].

“Harmonic structure”, consisting of a coarse-grained structure (“core”) and a three-dimensional continuously connected network of ultrafine-grained structure (“shell”), has been proposed by Ameyama [26] and others [27-39]. Commonly, the distribution of CG and UFG is random in the traditional bimodal structure, which may tend to induce strain localization. By contrast, according to the experiments and simulation of harmonic structure, the peculiar macroscopic network structure can restrict the strain localization effectively during plastic deformation, which results in an extra strain hardening [28, 29, 35]. A variety of harmonic structure designed materials, such as stainless steel [26-31], pure Ti [32, 33], Ti-6Al-4V [34, 35], Cu [36, 37] and Co-Cr-Mo [38, 39], demonstrated a good combination of high strength and large elongation, especially large uniform elongation.

The appropriate grain size and volume fraction for the design of the bimodal structured materials with high strength and high ductility have been investigated [40-44]. Hono et al. [42] reported that bimodal grain size distribution with nanocrystalline grains of 80 nm and coarse grains of nearly 1 μm in a ferrite solid solution demonstrated high compressive yield strength and large plasticity. Moreover, the appropriate volume fraction of the nanocrystalline grains for the bimodal structured Fe is approximately 60-65% [40]. A similar tendency was also reported in a bimodal structured Al/Mg alloy by Han et al. [43]. However, the desirable volume fraction of the ultrafine grains for the bimodal structured pure Ti is approximately 85% [44]. In the previous studies, the exact grain size and volume fraction of CG and UFG are quite different in the

harmonic structured materials. The outcome properties of the harmonic structured materials span a relatively wide range. So far, the optimized grain size and volume fraction of CG and UFG for harmonic structure designed materials have not been revealed yet.

In the previous studies [26-39], a planetary ball mill was applied to synthesize the bimodal structured powders before sintering. However, the grinding action will stop if the centrifugal force acting on the steel balls exceeds the force of gravity at a certain speed. So it was difficult to introduce very high energy to the powders by planetary ball mill. By contrast, horizontal ball mill has a horizontal shaft with a series of impellers inside container, which can rotate at high speed. Moreover, grinding action can occur between balls, between balls and container, and between balls, shaft, and impellers [45]. The horizontal ball mill can generate much higher energy than the planetary ball mill.

Powder metallurgy method can greatly reduce the amount of used metals compared to other metal removal processes, thereby reducing yield losses in manufacture and often resulting in lower costs. The harmonic structure designed stainless steel has high application value and development potential in medical industry. Therefore, the objective of the present study is to fabricate the harmonic structured SUS316L compacts by high energy ball milling and subsequent hot isostatic sintering process. Then, investigate their microstructure evolution and mechanical properties. Finally, try to reveal the appropriate microstructural characteristics, i.e. grain size and volume fraction of CG and UFG, for the harmonic structure designed austenitic stainless steel through analyzing the relationship between microstructure and mechanical properties.

2. Experiment

Commercial gas-atomized SUS316L powders were provided by Fukuda Metal Foil & Powder Co., Ltd. The powder particle size ranges from approximately 1 μm to 20 μm , and the chemical composition is shown in Table 1, which corresponds to the Japanese Industrial Standard (JIS G 4303: 2012). Firstly, initial powders were mechanical milled by a horizontal ball mill (Zoz Simoloyer CM01) under argon gas atmosphere at room temperature. SUS304L balls (5mm diameter) and container were used, and the ball-to-powder weight ratio was 8:1. The mechanical milling was carried out for 36ks, 108ks and 180ks at a constant speed of 1000 rpm, respectively. Then, the milled powders were charged into a mild steel capsule and sintered by hot isostatic pressing (HIP) with a pressure of 200MPa for 14.4ks at 1173K. The symbols IP, MM36ks, MM108ks, and MM180ks mean that the compacts sintered from the powders milled for 0 ks, 36 ks, 108 ks, and 180 ks, respectively.

Microstructure was observed by an electron backscattering diffraction (EBSD) system (FEI (Philips) Siron) operated at 15 kV. Grain size, high-angle grain boundaries (HAGBs) and low-angle grain boundaries (LAGBs) were mapped. The diameter of grains and area fraction of the “core” and “shell” regions were defined as grain size and volume fraction by calculating from images.

In order to study the Hall-Petch relationship in the harmonic structured SUS316L steels, the average grain size (d_A) of the entire microstructure is determined by an area (volume) fraction-the inverse grain sizes in the two regions, i.e. $d_A^{-1} = d_{\text{shell}}^{-1} \times S_f + d_{\text{core}}^{-1} \times (1-S_f)$. Where S_f is the volume fraction of the ultrafine-grained structure (shell), d_{shell} and d_{core} are the average grain size of ultrafine grains and coarse grains, respectively. The mentioned method has been applied in many papers to discuss the effects of grain size on mechanical properties of bimodal structured materials

[40, 46].

Mechanical properties, such as hardness, strength and elongation, were evaluated. The Vickers hardness was measured under a load of 0.98 N. The tensile specimens had a gauge length, width and thickness of 3mm, 1mm and 1mm, respectively. Tensile tests were carried out on a universal testing machine under displacement control with a nominal strain rate of 5.56×10^{-4} /s (cross-head speed of 0.1mm/min). The displacement of gauge area was recorded by a non-contact CCD camera system. At least four specimens were tested for each specimen condition, and the obtained results were reproducible.

3. Results and discussion

3.1 Microstructural characteristics of the harmonic structured SUS316L compacts

The morphology of initial powders and milled powders is shown in Fig.1. As shown in Fig.1a, the particle size of gas-atomized initial powders was irregular owing to the rapid cooling. The flake-shape powders were produced after mechanical milling for 36 ks and 108 ks (see Fig.1b and 1c). By contrast, the milled powders were agglomerated owing to cold-welding between the powder particles as the milling time increased to 180 ks (see Fig. 1d).

Figure 2 shows the microstructure of SUS316L stainless steels produced from various preparation methods. Fig.2a-2e are grain boundary maps, wherein HAGBs and LAGBs are drawn by blue lines and green lines, respectively. Fig.2f-2j are grain distribution maps, wherein the aqua blue area indicates the grain size is over than 1 μm , while the orange area means the grain size is less than 1 μm . As shown in Fig.2a and 2f, the common SUS316L bulk has a homogeneous CG microstructure with an average grain size of 15.6 μm .

It can be observed from Fig. 2b and 2g that a bimodal structure composed of coarse grains and ultrafine grains is formed in IP compact owing to the irregular particle size of initial powders. The coarse grains are surrounded by ultrafine grains. By contrast, an increase of milling time leads to a decreased grain size and an increased volume fraction of ultrafine grains (Fig.2c-e and Fig.2h-j). Moreover, as shown in Fig.2h-2j, it is notable that the clear connected network of ultrafine-grained structure can be observed. Namely, due to the wide distribution of particle size of initial powders, a partially harmonic structure is formed in the sintered compact without milling. Furthermore, the clear harmonic structure can be obtained in the sintered compacts prepared from milled powders.

The XRD results of the milled powders and sintered compacts are shown in Fig. 3. As shown in Fig.3a, both FCC and BCC structure can be observed in the initial powders. It is thought that the initial powders have both austenite phase and ferrite phase, which may be related to the rapid cooling during the atomization. By contrast, the peak of BCC structure increases gradually as milling time increasing, which indicates that the amount of ferrite phase in the vicinity of the milled powders surface increases gradually. It was reported that the nano ferrite grain structure can be formed in SUS316L powders by the milling process, which is referred to as a strain-induced-ferrite transformation [47]. However, it can be seen from Fig. 3b that both IP compact and the compact prepared from milled powders have FCC structure only, which indicates that all of ferrite phase transfers to austenite phase during sintering process.

Figure 4 and Table 2 present the variation of grain size and UFG volume fraction in the sintered compacts. The average grain size of the core in IP compact is approximately 4.0 μm , while the shell has an average grain size of approximately 1.5 μm . It can be noted that the average grain size in the core decreases gradually as the milling time increases. While, mechanical milling leads to a

minor change of grain size in the shell, and the grain size ranges from 0.2 μm to 1.0 μm . The shell fraction increases monotonically from 26.5% to 73%.

Figure 5 shows the variation of the average grain size (d_A) in the sintered compacts. Although grain coarsening should occur during the HIP, d_A still shows some correlation with milling time. Namely, d_A logarithmically decreases with the increase of milling time. Moreover, it is worth emphasizing that $d_A^{-1/2}$ increases linearly as the milling time increase. Therefore, it may be concluded that the desirable harmonic structured SUS316L stainless steel can be acquired by controlling milling time.

Mechanical milling is a well-known method for producing UFG or NG materials. In the previous study [27], the average grain size of shell was approximately 2 μm in the harmonic structured austenitic stainless steels produced by the planetary ball mill. Even the milling speed and time were attempted to be increased, the shell grain size was difficult to be lower than 1 μm . By contrast, much higher energy can be provided by the horizontal ball mill, which induces finer grain size in the present study.

3.2 Improvement of mechanical properties in the harmonic structured SUS316L compacts

The summarized tensile results of SUS316L stainless steels subjected to different types of processing are shown in Figure 6 and Table 2. Figure 6 shows the nominal strain- nominal stress curves of the specimens with different grain structures. As usual, the CG SUS316L bulk has good ductility but the strength is relatively low. The homogeneous UFG or NG SUS316L steels produced by cold rolling, SMAT [1] or thermal cycling [48-50,] always show extremely high strength (yield strength is over than 1000MPa), but the uniform elongation is inferior (uniform

elongation is lower than 2%). By contrast, it is noteworthy that the yield strength and ultimate tensile strength of the harmonic structured SUS316L compacts are much higher than those of the CG bulk. Furthermore, the harmonic structured SUS316L compacts also show higher strain hardening capability and larger uniform elongation compared to those of the homogeneous UFG or NG steels. Severe plastic deformation (SPD) followed by appropriate thermal annealing can be used to synthesize the bimodal structured SUS316L stainless steels. Even compared with the heterogeneous bimodal structured SUS316L steels produced by a combination of ECAP and annealing [51], the harmonic structured SUS316L steel still show a superior balance of high strength and high ductility.

The strain energy density (SED) of the sintered compacts is shown in Fig.7. The SED of IP compact is approximately 307 J/cm^3 . As the milling time increasing, the SED of the sintered compacts prepared from milled powders decreases gradually. The SED of MM180ks compact is only 70 J/cm^3 . The decreased SED indicates that the ductility of the SUS316L compacts prepared from milled powders decreases as milling time increasing.

Figure 8 shows comparison between the yield strength and uniform elongation of the SUS316L stainless steels having various microstructures. Although the IP compact has partially network ultrafine-grained structure, improved balance of strength and ductility can be achieved. Moreover, the harmonic structured SUS316L compact prepared from MM108ks powders shows a superior combination of high strength and improved uniform elongation ($\sigma_y=695\text{MPa}$, $\epsilon_u=27.1\%$).

Compared to the homogeneous UFG SUS316L steel produced by thermal cycling [50] with an average grain size of approximately $3 \mu\text{m}$, the harmonic structured compact show slightly inferior ductility, but nearly one time higher yield strength. By contrast, compared to the SUS316L steel

with a homogeneous grain size of approximately 1.4 μm , the harmonic structured steel has similar yield strength, but nearly one time larger uniform elongation. Compared with the heterogeneous bimodal structured SUS316L steels having similar CG size (approximately 1.0 μm) and CG volume fraction (approximately 40%) [51], the harmonic structured SUS316L steel (MM108ks) shows higher yield strength and larger uniform elongation. It is well-known that the inferior ductility of UFG or NG materials is caused by the early appearance of plastic strain instability [6-8, 16]. The difference of mechanical properties between harmonic structured stainless steel and heterogeneous bimodal structured one have been also studied [28]. It was found that both strength and ductility can be simultaneously increased in the harmonic structured steels. Moreover, the harmonic structure can thwarts the localization of plastic flow during mechanical loading. According to the simulation of tensile deformation behavior of the harmonic structured stainless steels, it is worth emphasizing that not only core can promote strain hardening capability, but also the peculiar network shell induces uniform distribution of strain during plastic deformation [29]. Therefore, the great balance of high strength and large uniform elongation can be achieved in harmonic structured SUS316L compacts.

3.3 Appropriate harmonic structure design for the austenitic stainless steels

It is well-known that the mechanical properties, i.e. hardness, strength and ductility, are related to grain size of metals [6-8]. Figure 9 shows the relationship between average grain size and mechanical properties. It can be observed that both hardness and yield strength increase linearly with the increase of $d_A^{-1/2}$. It has been reported that the yield strength resembled the Hall-Petch relationship in the homogeneous grain-structured SUS316L steels [1, 14]. Although the harmonic

structured SUS316L compacts have a bimodal grain size distribution, both hardness and yield strength still well resembles the Hall-Petch relationship.

In order to acquire the appropriate harmonic structure design, i.e. grain size and volume fraction of CG and UFG, for austenitic stainless steel. The effects of core grain size and shell fraction on the mechanical properties of the harmonic structured SUS316L compacts are discussed in Fig. 10 and Fig. 11, respectively. Because the variation of the shell grain size is not significant in the harmonic structured SUS316L compacts, so only the effect of core grain size on the mechanical properties is discussed here. It can be noted from Fig. 10a that both yield strength and ultimate tensile strength increase with the decrease of d_{core} , but the tendency of increment is different. Namely, the yield strength tends to close to ultimate tensile strength as d_{core} decreases to approximately 1.2 μm . As well as strength, uniform elongation and total elongation also demonstrate different monotonic behavior (see Fig. 10b). It is noteworthy that the uniform elongation decreases slightly as d_{core} decreases to 1.2 μm . A similar tendency has also been observed in a bimodal structured ferrite solid solution by Hono et al. [42]. Figure 11 presents the relationship between shell fraction and mechanical properties of the harmonic structured SUS316L compacts. The total elongation decreases monotonically from approximately 50% to 28% as the shell fraction increases to approximately 55%, while the uniform elongation decreases slightly. However, the decline of uniform elongation and total elongation is obvious as the shell fraction is more than 70%. Namely, the strength can be increased as the volume fraction of ultrafine grains continues to increase, but the balance of high strength and high ductility tends to be deteriorated.

Therefore, it is suggested that for the harmonic structure designed austenitic stainless steel, the core grain size of approximately 1.2 μm and shell volume fraction of approximately 50%-60% are

the optimized microstructure characteristics.

4. Conclusions

In summary, the harmonic structure designed SUS316L compacts were produced by the combination of horizontal ball milling and hot isostatic pressing. Microstructure evolution and mechanical properties of the harmonic structured SUS316L stainless steels were analyzed. The following conclusions can be obtained:

(1) Owing to the wide distribution of initial powder size, partially harmonic structure can be formed in the sintered compact without mechanical milling. The harmonic structure designed SUS316L compacts can be acquired by high energy ball milling and subsequent powders consolidation. The desirable microstructure can be controlled by adjusting milling parameters.

(2) A great combination of high strength and improved uniform elongation ($\sigma_y=695\text{MPa}$, $\epsilon_u=27.1\%$) can be achieved in the harmonic structured SUS316L compact. The improved ductility was attributed to both the strain hardening induced by the coarse-grained structure and the extra strain hardening caused by the peculiar macroscopic network of ultrafine-grained structure.

(3) Although the harmonic structured SUS316L compacts have a bimodal grain size distribution, both hardness and yield strength still well resembles the Hall-Petch relationship.

(4) The core grain size of approximately $1.2\text{ }\mu\text{m}$ and the shell volume fraction of approximately 50%-60% are recommended to be the optimized harmonic structure design for austenite stainless steel.

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Table Captions:

Table 1 Chemical composition of commercial SUS316L gas-atomized powders (mass%).

Table 2 Microstructural characteristics and mechanical properties of SUS316L stainless steels subjected to various preparation methods.

Figure captions:

Figure 1 The morphology of (a) Initial powders; (b) Powders milled for 36ks; (c) Powders milled for 108ks; (d) Powders milled for 180ks.

Figure 2 Microstructure of SUS316L stainless steels produced by different preparation methods: bulk (a and f), IP (b and g), MM36ks (c and h), MM108ks (d and i) and MM180ks (e and j). (a-e) are grain boundary maps. The blue lines indicate misorientations above 15° , and the green lines between 1 and 15° . (f-j) are grain size distribution maps. The aqua blue area indicates the grain is over than $1\mu\text{m}$, and the orange area is less than $1\mu\text{m}$.

Figure 3 The XRD results of (a) milled powders and (b) sintered compacts.

Figure 4 Effect of milling time on microstructural characteristics of the sintered SUS316L compacts: (a) Grain size; (b) UFG volume fraction.

Figure 5 Effect of mechanical milling time on average grain size of the sintered SUS316L

compacts.

Figure 6 Tensile results of SUS316L stainless steel specimens having various microstructures.

Figure 7 The strain energy density (SED) of the sintered compacts.

Figure 8 Relationship between yield strength and uniform elongation of SUS316L stainless steels having various microstructures.

Figure 9 Diagram illustrating the dependence of mechanical prosperities of the sintered SUS316L compacts on the average grain size: (a) Vickers hardness; (b) Yield strength.

Figure 10 Relationship between mechanical properties and core grain size in the harmonic structured SUS316L compacts: (a) Strength vs d_{core} ; (b) Elongation vs d_{core} .

Figure 11 Relationship between mechanical properties and shell fraction in the harmonic structured SUS316L compacts: (a) Strength vs S_f ; (b) Elongation vs S_f .

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Table 1 Chemical composition of SUS316L gas-atomized powders (mass%).

C	Si	Mn	P	S	Ni	Cr	Mo	Fe
0.018	0.9	1.07	0.032	0.017	12.44	17.31	2.11	Bal.

Table 2 Microstructural characteristics and mechanical properties of SUS316L stainless steels subjected to various preparation methods.

No.	Processing**	σ_y^* [MPa]	σ_{UTS}^* [MPa]	ϵ_u^* [%]	ϵ_t^* [%]	Average grain size [μm], (fraction %)		Ref.
						bulk or CG	UFG	
1	IP	470	693	31.0	48.0	3.94 (86.7%)	1.28 (13.3%)	Present
2	MM36ks	590	778	29.2	37.3	1.84 (73.5%)	0.64 (26.5%)	Present
3	MM108ks	695	802	27.1	28.2	1.16 (45.5%)	0.42 (54.5%)	Present
4	MM180ks	810	841	8.1	8.6	1.06 (26.5%)	0.33 (73.5%)	Present
5	Bulk	315	585	66.8	87.7	15.6 (100%)	---	Present
6	HR at 1173K	690	746	15.3	43.3	1.40 (100%)	---	Present
7	CR at 298K	1024	1376	1.25	20.3	0.76 (100%)	---	Present
8	SMAT	1450	1550	1.0	3.4	0.04 (100%)	---	[1]
9	15% CR at 77 K	680	1050	---	12	---	---	[48]
10	ECAP at 423K-1pass	850	900	15.0	30.0	---	---	[49]
11	ECAP at 423K-3pass	1300	1340	1.0	18.0	0.01-0.04 (100%)	---	[49]
12	85%CR HT at 1223K/3min	350	680	35.0	45.0	3.10 (100%)	---	[50]
13	ECAP + Annealing at 973K	640	880	18.0	---	1.0 (40%)	NT (60%)	[51]
14	ECAP + Annealing at 1073K	380	720	43.0	---	1.0-5.0 (90%)	NT (10%)	[51]

* σ_y –yield stress; σ_{UTS} –ultimate tensile stress; ϵ_u –uniform elongation; ϵ_t –total elongation.

** MM–mechanical milling; IP–initial powder; HR–hot rolling; CR–cold rolling; SMAT –surface mechanical attrition treatment; HIP – hot isostatic pressing; ECAP – equal channel angular pressing; and HT–heat treatment; and NT–nano twins.