**The deformation and fracture** **behaviors of 316L stainless steels fabricated by spark plasma sintering technique under uniaxial tension**

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**ABSTRACT**

In this study, 316L stainless steel (SS) specimens with different relative densities were fabricated using the spark plasma sintering (SPS) technique. These SPS specimens were used to capture the effect of microstructure heterogeneity on deformation and fracture behaviors during uniaxial tension. Microstructure analysis indicated that the SPS specimens consisted of fully sintered and partially sintered regions and contained initial pores which are located at the grain boundaries. Mini-tension tests combined with the digital image correlation (DIC) technique were carried out at room temperature to measure the mechanical properties of the SPS specimens and the evolution of strain heterogeneity on tensile specimens during uniaxial tension. In order to reveal the fracture mechanisms of the SPS specimens, the surfaces of the fractured specimens were analyzed via field emission scanning electron microscope (FE-SEM). The fracture mechanism in the fully sintered region was identified as a ductile fracture by the formation of cup-like dimples, while the fracture mechanism in the partially sintered region was identified as a decohesion of the interface between the powder and the matrix.

**Keywords:** 316L stainless steel, SPS, DIC, Microstructure, Fracture.

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1. **Introduction**

~~As a structural material,~~ 316L austenitic stainless steel ~~(SS)~~ has been extensively used as a structural material ~~is an industry standard~~ due to their excellent properties such as high-temperature oxidation resistance, high resistance to corrosion and abrasion, high mechanical strength, and excellent formability and weldability [1,2]. Recently, the use of 316L SS has expanded to the petrochemical industry [3], the medical sector [4,5], and the nuclear industry [6]. However, prior studies have shown that 316L SS suffers severe irradiation damage even within a moderate temperature range of 150~400 °C [7–9]. Research shows that refinement of the grain size of 316L SS can reduce the radiation-induced segregation at the grain boundaries [10,11], which in turns prevents early failure. An effective selection of processing conditions is essential to improve the mechanical properties and corrosion resistance of SS in order to expand its range of industrial applications and usage under adverse environmental conditions [2,12–16]. Considerable research has been conducted to improve the mechanical strength as well as irradiation resistance of ultra-fine-grained or nanocrystalline 316L SS by optimization of its thermo-mechanical processing conditions [17,18]. Several processing techniques have been developed to produce near-net shaped parts: powder injection molding ~~(PIM)~~ [19–21], powder metallurgy ~~(PM)~~ [22], and spark plasma sintering ~~(SPS)~~ [1,2,15,23].

Among these ~~processes, high speed~~ rapid powder sintering process~~, i.e.~~ SPS has been widely used for metallic materials and ceramics due to its unique operating conditions and processing advantages such as relatively low-temperature processing, high material utilization (95%), process-induced microstructure tailoring ability, and short sintering times [24–29]. In the SPS process, the volumetric heating rate resulting from the Joule effect induces a rapid increase in the temperature, which improves the consolidation rate [23]. Many studies on SPS have been conducted on various alloys, such as Fe-Ni [30,31], pure Nickel [32,33], Ti alloys [34–36], Cobalt [37], and steel [13,14,38,39]. However, few studies have been focussed on the effect that the relative density of 316L SS fabricated by SPS exerts on deformation and fracture behaviors during uniaxial tensile testing. Hardness measurements are insufficient for a characterization of the mechanical properties of sintered samples, e.g. ductility [1,2]. No detailed analysis has addressed the correlations between the microstructure and the mechanical properties of the 316L SS fabricated by SPS.

The purpose of the present study was to understand the deformation and fracture behaviors of 316L SS fabricated by SPS under uniaxial tension. Specimens with different relative densities were prepared using the SPS process. In order to understand the deformation behavior, local strain distributions in different specimens were examined using the DIC technique. Finally, systematic fracture analysis were performed both on the fracture surface and perpendicular to the fracture surface to understand the mechanism of failure for SPS specimens with different relative densities.

1. **Experimental**

*2.1. Spark plasma sintering*

Commercially available 316L SS powder (CL 20ES [40]) was used for this work. The chemical composition of the CL 20ES powder used for the SPS processing is shown in Table 1. The powder morphology and microstructure were analyzed using an FE-SEM. The shape of the powders was mostly spherical, as shown in Fig. 1(a), with diameters that varied from 1.1 ~ 79.1 μm (Fig. 1(b)). The average diameter of the powder was established at 7.3 μm. The presence of oxides on the surface of the constituent particles was confirmed via energy dispersive spectroscopy (EDS) analysis, as shown in Fig. 1(c). The as-received powders were pressed in a graphite die 50 mm in diameter and placed in between the upper and lower electrodes of an SPS apparatus. SPS processing mainly depends on four main parameters: heating rate, soaking temperature, applied pressure, and holding time. Fig. 2(a) shows a typical schematic diagram of the SPS apparatus with a vertical uniaxial pressure system (upper and lower electrode). Powders were heated up by applying a pulsed electric current. The temperature was monitored by an infrared pyrometer focused onto the inside of ~~through a hole in~~  the upper graphite punch at a distance of ~~at~~ about 4 mm from the sample. Sintering was performed by applying a uniaxial pressure under vacuum. The powders were separated from the die and punches using graphite foils to prevent sticking during the process. SPS processing was performed using two different conditions (SPS-I and SPS-II) in terms of heating rate and soaking temperature, for a fixed soaking time of 5 min, as shown in Table 2. The processing conditions for the SPS-I specimen (heating rate: 100 °C/min, soaking temperature: 1100 °C) were selected according to optimum conditions suggested by Marnier *et al.* [1], as shown in Fig. 2(b). A heating rate of 200° C/min and a soaking temperature of 1,025 °C were employed for the SPS-II specimen, as shown in Fig. 2(c). The density of the specimens was measured via the Archimedes method (RADWAG-AS-R-series) [41]. The chemical composition of the SPS specimen is described in Table 1, whereas the relative densities of the SPS-I and SPS-II specimens are described in Table 2.

* 1. *Microstructure characterization and tensile test*

Microstructural analysis of the SPS specimens was conducted on the section normal to the first lateral direction (LD1). As-fabricated specimens were mechanically polished from P2400 SiC paper to colloidal silica of 0.04 μm. Polished specimens were cleaned ultrasonically with ethanol and then dried by blowing hot air. Prior to observing the optical microstructure, all specimens were chemically etched by Marble’s reagent (10 g CuSO4, 50 ml HCl, and 50 ml distilled water) for 50-60 sec. Microstructures were then observed using an optical microscope (OLYMPUS GX-51).

The microtexture of the SPS specimens was examined via electron backscattered diffraction (EBSD) analysis with a ~~field emission scanning electron microscope (~~FE-SEM~~)~~ (JEOL, JSM-7100F). After observation of optical microstructures, mechanical polishing was conducted using a 1 μm diamond paste for the intermediate stage. Finally, the specimens were polished using 0.04 μm colloidal silica. The EBSD analysis was examined by selecting a scanning area of 300 × 300 μm2 at a step size of 0.5 μm. Microtexture examination was performed using TSL-OIMTM (TexSEM Laboratories orientation imaging microscopy) software along with techniques developed specifically for analyzing twin boundaries, grain size distribution, and kernel average misorientation (KAM) distribution.

Mechanical properties were measured using a mini-tension tester combined with a DIC technique, as shown in Fig. 3(a). Fig. 3(b) is a schematic of how the miniature specimen was machined from the as-fabricated SPS specimen. CD, LD1, and LD2 represent the compression direction, first lateral direction and second lateral direction (perpendicular to LD1), respectively. Tensile testing was performed at room temperature with a strain rate of 0.001/s. The values for total length and parallel gauge length of the miniature tensile specimens were 18 mm and 9 mm, respectively. The values for width and thickness of the parallel gauge length were 2 mm and 0.8 mm along the LD1 and CD, respectively, as shown in Fig. 3(c). The fracture surface of the fractured tensile specimens was examined using an FE-SEM (JEOL, JSM-7100F) on the LD2 section (i.e., on the fracture surface). To examine the crack initiation mechanism, additional analysis on fractured specimens was conducted on the cross-sectional plane perpendicular to the fracture surface (i.e. LD1 section). An EDS line scan in tandem with the FE-SEM analysis was performed perpendicular to the fracture surface to determine the chemical composition of the specimens at the fracture sites.

1. **Results and Discussion**

Optical and FE-SEM images of the SPS specimens measured in the LD1 section are shown in the Fig. 4. The optical microstructures indicate that the SPS specimens exhibited a bimodal grain size distribution consisting of fully sintered and partially sintered regions, regardless of the SPS process conditions. No noticeable differences could be found in the optical microstructures of the SPS specimens, as shown in the Figs. 4(a) and (c). Figs. 4(b) and (d) are FE-SEM images showing the surface morphologies of the SPS specimens. The SPS-I specimen contained pores at grain boundaries that were homogeneously distributed and relatively fine, as shown in the *inset* of Fig. 4(b). The SPS-II specimen, however, contained a relatively high fraction of coarse pores at the grain boundaries, as shown in the *inset* of Fig. 4(d). This result indicates that the porosity of the SPS specimens was decreased with increases in the processing temperature, but only if the effect of the heating rate is disregarded. However, it is known that lowering the heating rate is favorable for decreasing the porosity in the SPS specimens [42].

Fig. 5 shows the inverse pole figure (IPF), image quality (IQ), and KAM maps that were analyzed for the LD1 section of the SPS specimens. The color code of the IPF map represents the crystallographic orientation parallel to the LD1. The IPF maps in Figs. 5(a) and (d) indicate that the grains in the SPS specimens were randomly oriented with no preferred orientation with respect to the sample coordinates. The calculated IPF, as shown in Fig. 6(a), indicates that the SPS-I and SPS-II specimens had an almost random orientation. Figs. 5(b) and (e) show the distribution of the high-angle grain boundaries (HAGBs) and the Σ3 twin boundaries (TBs) in the IQ maps of the SPS specimens. The blue, and red lines in the IQ maps represent the HAGBs with a boundary misorientation of 15° to 65°, and Σ3 TBs, respectively. The length fractions of the grain boundaries (GBs) are listed in Table 3. The tolerance angle for the angular and twin-plane to distinguish the Σ3 TBs was 5° [43]. These results indicate that the GBs of the as-fabricated SPS specimens consisted of a large fraction of HAGBs and a small fraction of Σ3 TBs. A higher fraction of Σ3 TBs was found in the SPS-I specimen compared with that of the SPS-II specimen. Thus, increasing the processing temperature (i.e. soaking temperature) allowed the percentage of Σ3 TBs to increase [1].

The porosity was directly related to extremely low IQ regions within the microstructure, as shown in Figs. 5(b) and (e). The SPS-II specimen contained relatively larger regions of low IQ compared with the SPS-I specimen, as shown in Fig. 5(e), which also confirmed the presence of a relatively high fraction of initial pores, as shown in Fig. 4(d). Clustering of the pores was found in the SPS-II specimens. EBSD analysis of the misorientation angle distributions, as shown in Fig. 6(b), indicated that the SPS specimens exhibited a high fraction of misorientation around 60° that was due mainly to the Σ3 TBs. KAM maps based on the 3rd nearest neighbors of each point, and with an upper limit of 5o, indicated the degree of plastic deformation in the material. The regions with a relatively high KAM value, as shown in Figs. 5(c) and (f), seemed related to a remaining deformed regions even after SPS processing. In the KAM map, the SPS-II specimen showed a relatively stronger KAM distribution than that of the SPS-I specimen. These results explain why the SPS-II specimen had a relatively large deformed portion compared with the SPS-I specimen, as shown in Fig. 5(f). The grain size and KAM distributions of the SPS specimens are shown in Figs. 6(c) and (d), respectively. The area fraction of the grain size indicates that the SPS-I specimen had a low fraction that was in the range of a small grain size compared with that of the SPS-II specimen, as shown in Fig. 6(c). The SPS-II specimen exhibited a fraction that was relatively high and in the range of KAM above 1°, as shown in Fig. 6(d). This observation based on KAM ~~result~~ indicates that the SPS-II specimen contained ~~a~~ relatively higher fraction of deformed regions than that of SPS-I specimen.

Fig. 7 shows the deformation behavior of the SPS specimens monitored by DIC during tensile testing. The 2-D strain maps were constructed by utilizing the digital images captured at different stages of the tensile testing and using the ARAMIS software [44], as shown in Figs. 7(a), and (b). Figs. 7(c), and (d) represent the corresponding strain distributions along the line profile of the gauge section under various global engineering strains. The plastic strain was relatively homogeneously distributed in the deformed SPS specimens until a global engineering strain of 0.075, while the SPS specimens exhibited a distinct strain heterogeneity from a global engineering strain of 0.09 before failure. The SPS-II specimen had a relatively large number of locally deformed regions in the tensile specimen from a global engineering strain of 0.09, as shown in Fig. 7(b). It seems that the relatively low density of the SPS-II specimen was closely related to the distinct development of the locally deformed regions in the tensile specimen during uniaxial tension.

Fig. 8 compares the mechanical properties of the SPS specimens. The average tensile strengths of the SPS-I and SPS-II specimens were 681 MPa and 609 MPa, respectively. The ductility of the SPS-II specimen was slightly lower than that of the SPS-I specimen. That result seemed to be due to a relatively high fraction of initial pores in the SPS-II specimen [45]. As the sintering temperature increased, we found that both the strength and ductility of the specimens could be increased [46]. The SPS specimens exhibited a relatively high yield strength and a low level of ductility compared with that of compacts sintered in nitrogen and argon [47]. Due to the presence of oxides on the surface of the constituent particles (Fig. 1(c)), yield strength could further increase [2], which would consequently decrease the sample ductility [48]. The initial pores inside the SPS specimens seemed to be contributed to the strain localization in the deformed specimen during uniaxial tension. Static mechanical properties such as strength and ductility were found to be greatly affected by the initial pores. The SPS-I specimen, which had a relatively low fraction of initial pores, exhibited better mechanical properties.

The instantaneous strain hardening rate, (d/d) was analyzed using the true stress- strain curves of the SPS-I and SPS-II specimens, as shown in Figs. 8(c) and (d). The d/d was drastically decreased during the initial plastic deformation. Then, after passing a narrow transition region, the d/d gradually decreased as strain increased. It should be noted that the value of d/d after the initial yielding was similar in both SPS specimens. Moreover, the SPS specimens showed no post-elongation following finishing of uniform elongation. As shown in Fig. 7, strain concentration (highlighted by more brighter color (e.g., yellow or red)) occurred primarily at the initial pores, which could have produced the negligible post-elongation.

FE-SEM analysis was conducted to identify the main reasons for the negligible post-elongation of the SPS specimens in detail. Fig. 9 shows the fractured surface of the SPS-I specimen. This result shows that both partially sintered and completely sintered regions exhibited different surface morphologies. The fractured surface of the completely sintered region consisted of the formation of fine cup-like dimples, which indicated ductile failure, as shown in the upper FE-SEM images (a1–a3) of Fig. 9. The fracture surface of the partially sintered region consisted of ~~incomplete dimples, which indicated~~ weak bondings between the powder and the matrix, as shown in the lower FE-SEM images (b1–b3) of Fig. 9. Fig. 10 shows the fractured surface of the SPS-II specimen. The fracture surface of the SPS-II specimen was similar to that of the SPS-I specimen. The fractured surface of the completely sintered region consisted of fine cup-like dimples, as shown in the upper FE-SEM images (a1–a3) of Fig. 10. On the other hand, the lower FE-SEM images (b1–b3) of Fig. 10 explain that the fracture surface of the partially sintered region consisted of ~~incomplete dimples~~ region consisting of dimples and prior powder boundaries structures. The surface morphology of the partially sintered regions explains why the SPS specimens exhibited negligible post-elongation during uniaxial tension.

In order to capture the fracture mechanisms in the SPS specimens under uniaxial tension, additional FE-SEM images were taken in a cross-sectional plane which is perpendicular to the fracture surface for both the SPS-I and SPS-II specimens, as shown in Figs. 11(a) and (b), respectively. As shown in the upper FE-SEM images of Figs. 9 and 10, the ductile fractures were dominant in the completely sintered regions. However, during the SPS process, all of the constituent powder particles could not be properly sintered to form sintering necks, and thus the SPS specimens contained a low fraction of initial pores (Figs. 4(b) and (d)), which acted as sites for stress concentration under uniaxial tension. Figs. 11(a) and (b) show typical sites for crack initiation in the SPS specimens under uniaxial tension. Crack initiation occurred mainly at the interface between powder and matrix in the partially sintered regions. Decohesion at the interface seemed to play a significant role in the stress concentration in the fully sintered region of the SPS specimens during uniaxial tension. When tensile load was increased, the evolution of voids in the fully sintered region were promoted, and a debonding area at the interface between powder and matrix can accelerate the growth and coalescence of voids along an approximately vertical direction with respect to the loading direction. When sintering temperature is increased and the heating rate is lowered, the formation of sintering necks is well developed, and the density of the materials increases [42]. Relatively fewer of the initial pores in the SPS-I specimen seemed to contribute to higher ductility and enhanced mechanical strength, as shown in Fig. 8. The inset of Fig. 11 (b3) shows the ~~SEM–~~EDS line scan analyses, which were performed at the locations marked in Fig. 11 (b3). A distinct boundary between the matrix and the oxide layer appears in the cross-sectional FE-SEM image. The ~~SEM–~~EDS line scan analyses revealed that according to the oxygen distributions, which confirmed the presence of the oxide layer on the surface of the constituent particles, as shown in the Fig. 1(c).

Fig. 12 schematically explains the mechanism of decohesion at the interface between the powder and the matrix in the partially sintered region under uniaxial tension. Typical SPS specimens consist of both partially sintered and fully sintered regions and contain initial pores located at the grain boundaries, as shown in Fig. 12(a). With the application of a uniaxial tensile load, inhomogeneous deformation occurs to satisfy strain compatibility in the neighboring grains. However, the interface between powder and matrix in the partially sintered region cannot properly maintain continuity when the normal stress acting on the interface reaches a specific level and decohesion mainly occurs at interface approximately vertical to the loading direction, as shown in Fig. 12(b). Figs. 12(b2) and (b3) show the experimental evidence of the typical failure mechanisms that occurred in the SPS specimens during uniaxial tension. Fig. 12(c) shows the typical sites for void initiation in SPS specimens around the interface with precipitates (PPTs) or with the 2nd phase particles under uniaxial tension. As the tensile load increased, coalescence occurred to form large voids (Fig. 12(d)), and these large voids, together with the fractured surfaces propagated from the debonding area at the powder and matrix interface, caused failures in the SPS specimens, as shown in Fig. 12(e).

1. **Conclusions**

The ~~spark plasma sintering (~~SPS~~)~~ technique was used to fabricate 316L SS specimens with different relative densities. The microstructure of the SPS specimens consisted of both fully sintered and partially sintered regions and contained initial pores at the grain boundaries. As the sintering temperature increased under the assumption that the heating rate was negligible, relatively high density SPS specimens were produced. The relatively coarse initial pores in the SPS-II specimen contributed to the enhancement of KAM in the neighboring regions of pores. The increase in ultimate tensile strength and total elongation of the SPS-I specimen seemed to be due to the relatively low fraction of initial pores and to a lower degree of pore clustering, which produced locally deformed regions. DIC results indicated that strain concentration occurred primarily at the initial pores, which could have hindered the post-elongation. FE-SEM analysis of the fracture surfaces of the SPS specimens revealed that fine cup-like dimples were observed in the completely sintered regions, while ~~incomplete dimples~~ regions consisting of dimples and prior powder boundaries were observed in the partially sintered regions. Additional FE-SEM analysis of a cross-sectional plane, which was perpendicular to the fracture surface, revealed that crack initiation occurred mainly at the interface between powder and matrix in the partially sintered region under uniaxial tension. Decohesion at the interface was identified as the main fracture mechanism in the partially sintered region of the SPS specimens during uniaxial tension.

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**Table. 1**

Chemical composition of materials (wt %).

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **316L** | **Fe** | **C** | **Cr** | **Ni** | **Mo** | **Mn** | **Si** | **P** | **S** |
| CL 20ES [40] | Bal. | 0~0.03 | 16.5~18.5 | 10~14 | 2.0~3.0 | 0~2.0 | 0~1.0 | 0~0.045 | 0~0.03 |
| SPS | Bal. | 0.05 | 16.48 | 10.53 | 2.72 | 0.81 | 0.22 | 0.002 | 0.02 |

**Table. 2**

Processing conditions for fabrication of SPS specimens and corresponding densities.

|  |  |  |
| --- | --- | --- |
| **Parameters** | **SPS-I** | **SPS-II** |
| Soaking temp (°C) | 1100 | 1025 |
| Heating rate (°C/min) | 100 | 200 |
| Max. pressure (MPa) | 50 | 50 |
| Relative density (%) | 99.11 | 97.42 |

**Table. 3**

Length fraction of HAGBs and TBs.

|  |  |  |
| --- | --- | --- |
| **GBs** | **SPS-I** | **SPS-II** |
| HAGBs | 0.954 | 0.954 |
| TBs | 0.281 | 0.235 |

**Figure Captions**

**Fig.1.** (a) FE-SEM micrographs of as-received commercial 316L SS powder, and (b) corresponding powder size distribution. The EDS analysis shown in the panel (c) indicates the presence of oxides on the surface of the constituent powders.

**Fig. 2.** (a) Schematic of the SPS apparatus, the temperature and pressure cycles for the fabrication of (b) SPS-I (heating rate 100°C/min and soaking temperature 1100°C), and (c) SPS-II (heating rate 200°C/min and soaking temperature 1025°C) specimens.

**Fig. 3.** (a) Mini-tension tester, (b) schematic of miniature tensile specimens manufactured from as-fabricated SPS specimens, and (c) dimensions of the miniature tensile specimen cut from a bulk sample.

**Fig. 4.** The microstructure of (a,b) SPS-I and (c,d) SPS-II specimens: (a,c) optical and (b,d) FE-SEM images. The *inset*s of (b,d) show the partially melted powders in the SPS-I and SPS-II specimens, respectively. The arrows show the presence of the initial pores.

**Fig. 5.** EBSD measurement results of (a–c) SPS-I and (d–f) SPS-II: (a,d) IPF maps, (b,e) IQ maps, and (c,f) KAM maps. The arrows show the presence of the initial pores in the SPS specimens.

**Fig. 6.** (a) IPF, (b) distribution of the misorientation angles, (c) grain size distribution histogram, and (d) KAM distribution considering the 3rd nearest neighbors of SPS-I, and SPS-II specimens.

**Fig. 7.** Surface strain distribution maps resulting from the DIC measurement of (a) SPS-I and (b) SPS-II specimen for different applied engineering strain. Strain profiles along the center of the (c) SPS-I, and (d) SPS-II specimen at various applied engineering strains.

**Fig. 8.** Engineering stress–strain curves for (a) SPS-I and (b) SPS-II specimens. True stress-strain curves and work-hardening behavior of (c) SPS-I and (d) SPS-II specimens.

**Fig. 9.** FE-SEM fractography of the fractured surface of the SPS-I specimen after the uniaxial tensile test. The hierarchical FE-SEM images showing the presence of (a1–a3) completely and (b1–b3) partially sintered regions.

**Fig. 10.** FE-SEM fractography of the fractured surface of the SPS-II specimen after the uniaxial tensile test. The hierarchical FE-SEM images showing the presence of (a1–a3) completely and (b1–b3) partially sintered regions.

**Fig. 11.** FE-SEM cross-sectional (LD1 section, i.e. perpendicular to the fractured surface) view of the (a1–a3) SPS-I and (b1–b3) SPS-II specimens after the uniaxial tensile test. *Inset* of (b3) shows the ~~SEM-~~EDS line profile measured across the interface marked by a yellow line in the panel (b3).

**Fig. 12.** Schematics of (a) undeformed and (b1) deformed SPS specimens. (b2–b3) Experimental evidence of the typical failure that occurred in the SPS specimens during uniaxial tensile loading. Schematics of (c) void initiation, (d) void coalescence and (e) fracture of SPS specimens during uniaxial tensile loading.

**Figures**



