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Hardened austenite steel with columnar sub-grain structure formed by laser melting

K. Saeidi, X. Gao, Y. Zhong, Z.J. Shen *

Department of Materials and Environmental Chemistry, Arrhenius Laboratory, Stockholm University, S-106 91 Stockholm, Sweden

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ABSTRACT

Laser melting (LM), with a focused Nd: YAG laser beam, was used to form solid bodies from a 316L austenite stainless steel powder. The microstructure, phase content and texture of the LM stainless steel were characterized and compared with conventional 316L stainless steel. The crack-free LM samples achieved a relative density of 98.6 ± 0.1%. The XRD pattern revealed a single phase Austenite with preferential crystallite growth along the (100) plane and an orientation degree of 0.84 on the building surface. A fine columnar sub-grain structure of size 0.5 μm was observed inside each individual large grain of single-crystal nature and with grain sizes in the range of 10–100 μm. Molybdenum was found to be enriched at the sub-grain boundaries accompanied with high dislocation concentrations. It was proposed that such a sub-grain structure is formed by the compositional fluctuation due to the slow kinetics of homogeneous alloying of large Mo atoms during rapid solidification. The local enrichment of misplaced Mo in the Austenite lattice induced a network of dislocation tangling, which would retard or even block the migration of newly formed dislocations under indentation force, turning otherwise a soft Austenite to hardened steel. In addition, local formation of spherical nano-inclusions of an amorphous chromium-containing silicate was observed. The origin and the implications of the formation of such oxide nano-inclusions were discussed.

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1. Introduction

Stainless steel contains more than 10 wt% chromium and several other alloying elements like nickel and molybdenum. A popular grade 316L holds, besides iron, 17, 11 and 2 wt% of Cr, Ni and Mo, respectively. It contains only the austenite phase and is one of the most widely used stainless steels due to its good ductility, high strength, high corrosion resistance and relatively low cost [1–4]. Thus, 316L have a wide range of applications in oil and gas industry, refinery, chemical and petrochemical plants, constructions, automobile and recently for building components in nuclear reactors [5–7]. Production of stainless steel parts via powder metallurgy (PM) typically involves low to medium temperature solid state sintering. Consequently, the sintered compacts always contain residual porosity, which restricts their applications [8–9]. On the other hand, fabrication of highly dense stainless steel is of great demand. Performing consolidation at higher sintering temperatures through liquid phase sintering has shown to be promising in fabrication of fully dense 316L parts [10]. The presence of a liquid phase enhances densification by promoting capillary force induced pore filling besides interfacial diffusion. Liquid phase sintering requires high temperatures from 1300–1400 °C (depending on composition), which can hardly be facilitated by conventional sintering without a simultaneous grain growth. The microstructure coarsening might deteriorate mechanical properties. Sintering at high temperatures and avoiding grain growth is achievable, however, through the use of a high-energy focused laser beam and very short times at elevated temperatures.

Laser melting (LM) or laser sintering (LS) is a technique based on layer-wise building principle. It belongs to the group of additive manufacturing techniques. The difference between laser melting and commonly quoted laser sintering is slim; the first method refers to a fully melted laser spot volume, whereas the latter might comprise a melt and/or solids. Both have the potential to fabricate parts with complex geometry using a wide range of materials in short time and without the need of expensive tooling and machining. The energy is supplied by a focused laser beam whose scanning pattern is controlled by a computer based on CAD/CAM principle. More details about the setup of the LM process, the influencing parameters, advantages and disadvantages of the process and the potential applications of this technique can be found elsewhere [11–13].

In the past decade, laser melting of different kinds of metallic alloys have been developed and commercial products have been fabricated [14–17]. Recently several attempts on laser assisted fabrication of 316L stainless steel have been reported. The influences

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* Corresponding author. Tel.: +46 8 162388; fax: +46 8 152187.
E-mail address: shen@mmk.su.se (Z.J. Shen).

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of pulse frequency and duration of a pulsed wave CO_2 laser upon the microstructure, surface roughness and hardness of fabricated 316L have been studied by Pinkerton and Li [18]. The effect of laser parameters on balling phenomenon in a laser sintered 316L was investigated by Gu and Shen [19]. Laser irradiation of a 316L stainless steel coated with Ti and Si_3N_4 to improve the surface hardening was studied by Viswanathan [20]. The surface quality of LS 316L parts exposed to laser re-scanning was observed by Yasa and Kruth [21]. The cavitation erosion resistance of laser sintered mixture of stainless steel and NiTi powder was studied by Chiua et al. [22]. Finally, the pitting corrosion and sensitization in laser sintered 316L specimens was investigated by Ganesh [23]. Generally, all these reports on laser sintering of 316L have investigated the effects of the precursor material characteristics (such as chemical constitution, particle shape, size, and its distribution) and the laser processing parameters (such as laser power, scan speed, and scan line spacing) on the performance of the laser sintering/melting process and the resulting quality and properties of the parts. The microstructural evolutions of 316L during laser sintering/melting in comparison with conventional casting and welding, however, has not yet been studied in sufficient details.

Conventional melt-solidification during the casting and welding of 316L generally produces dendritic or columnar solidification-microstructures, depending on the degree of undercooling and the cooling rate. The correspondingly formed columnar grains in casted 316L steel are large, typically with size between 20 and 40 μm. Each columnar grain is accounted as a single crystal [24–25]. The size of columnar grains in solidification-microstructure formed by welding of 316L steel decreases to almost half the size in comparison with the casted samples due to a much higher degree of undercooling, but still many times larger than laser treated 316L. This fine columnar structure in laser treated 316L has also been observed and reported by others [5,17]. Columnar solidification-microstructures have not only been observed in 316L but also been seen in 304L stainless steel [24] and maraging 300 series steel prepared by a laser technique [26].

AISI 316L is extremely difficult to heat-treat for hardness due to its low carbon content. Carbon enrichment and surface alloying methods are therefore sometimes implemented before heat treatment operations. Formation of complex and non-homogeneous microstructure obtained by laser technique could result in hardened 316L steel.

The aim of the present work is to carefully examine the phase or structural changes and the microstructural evolution of the laser melted 316L stainless steel in terms of phase transformation, compositional gradients, microstructural changes and texture and the overall effect of these parameters on the hardness of the LM steel. Such information is essential for ensuring the quality and reliability of LM 316L steel parts.

2. Experimental methods and materials

Nitrogen-gas atomized spherical 316L steel powder (supplied by Sandvik Osprey Ltd., Neath, UK) with particle size of 22–53 μm was used as starting material. The overall chemical composition of the as-received powder provided by the manufacturer is shown in Table 1. Laser melting was performed in an Ar atmosphere of 1 bar with a residual oxygen partial pressure of 0.1%.

Laser melting was performed by using an EOSINT M 270 laser sintering facility (EOS, Krailling, Germany). A 200 W continuous wave Nd:YAG fiber laser operating at a wavelength of 1060 nm and with a typical focal spot size of 70 μm was used. The high power laser beam fuses the powder granules evenly placed on a flat building plate under the guide of a pre-determined pattern formulated by computer aided design (CAD). After complete exposing to the laser beam the building plate is lowered by 0.02 mm and a new even layer of powder granule is loaded on its top. The process is automatically repeated in a layer by layer manner until the part is built. Fixed laser parameters (power of 195 W, scan speed of 800 mm/s and line spacing of 0.1 mm) were used. These parameters have been optimized to obtain the highest density and to avoid surface defects (such as balling). The scanning was bi-directional and the angle of the scanning directions between each layer was chosen 60° for all of the samples. Dimensions of the test parts built were bodies of 5 × 5 × 2 mm and the parts built were detached from the building plate using wire electrical discharge machining (EDM). The densities of the prepared samples were measured by the Archimedes method.

Table 1
Overall chemical composition of the 316L powder (in wt%).

C	Mn	P	Cr	Ni	Mo	Si	O	N	Fe
0.01	0.98	0.02	17	10.6	2.3	0.4	0.05	0.15	Bal

X-ray diffraction (XRD) pattern was obtained using CuK_α radiation in a Panalytical XPert alpha1 diffractometer over a 2θ range between 30° and 100°. The JCPDS-card 31-0619 was used for identification of Austenite.

Macro- and microstructure observations were carried out by an optical microscope (OM) and a scanning electron microscope (SEM, JSM-7000F, JEOL, Tokyo, Japan). For even deeper microstructure observations transmission electron microscopes (TEM, FEG-2100F and LaB6 2100, JEOL, Tokyo, Japan) were used. For EDS mapping and line scan the FEG-2100F microscope (JEOL, Tokyo, Japan) was used. For line scan a length of 250 nm contained of 100 spots of 1.5 nm in diameter was chosen. Electron diffraction patterns (EDP) were obtained by a TEM 3010 microscope (JEOL, Tokyo, Japan). Before SEM observations the samples were mechanically polished and chemically etched in an acidic water solution containing 2% HF and 8% HNO_3 for 20 min at room temperature. For OM imaging the samples were only gently polished, but for SEM observations they were thoroughly polished. TEM samples were prepared by first grinding down the sample to approximately 150 μm thickness and thereafter further thinned down to electron transparency using Jet-polishing TenuPol-5 device (Struers, Ballerup, Denmark). The potential for jet-polishing was set to 25 V, the electrolyte was 15 vol% perchloric acid in methanol and the temperature was maintained at −60 °C. The texture of the prepared specimens were characterized by TESCAN MIRA 3LMH scanning electron microscope (TESCAN, Brno, Czech Republic) equipped with a HKL Nordlys orientation imaging microscope system for determining electron backscattered diffraction (EBSD),(HKL Technology, Hobro, Denmark). The EBSD data were processed by HKL Channel 5 software packages with a step size of 1 μm (Oxford Instruments, Oxford, UK). The cross section polishing required for EBSD was accomplished using an argon ion beam polisher SM-09010 (JEOL, Tokyo, Japan) under the accelerating voltage of 5 KV for seven hours.

The Logtering factor (LF) was used to quantify the degree of orientation for LM samples using Equation below [27,28].

$$LF = (P - P_0)/(1 - P_0) \quad (1)$$

where the calculated LF is the fraction of XRD peak areas obtained for a specific crystallographic plane. The term (P) is the fraction of the summation of all the peak intensities corresponding to the preferred orientation axis of the studied material. P_0 is the fraction of the summation of the peak intensities corresponding to the material with a random particle distribution (see corresponding JCPDS-card). The diffraction angle, 2θ, used for calculating the P (hkl) values is in the range of 40–100°. The LF varies between zero

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