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Novel ferritic stainless steel formed by laser melting from duplex stainless steel powder with advanced mechanical properties and high ductility

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ABSTRACT

Stainless steel bodies with relative density of 99.5% (with the theoretical density being 7.8 gr/cm^3) were manufactured by laser melting (LM) of duplex 2507SAF steel powder. The crystalline phases of starting powder were fully ferrite with only a small trace of austenite. The chemical composition was unchanged during laser melting. A unique mosaic-type structure with mosaics of 100–150 μm size was formed after LM. Recrystallized grains with 1–5 μm was formed in between the mosaic boundaries. A great number of entangled dislocation loops resembling a loops with 100–200 nm size were also formed inside each of these mosaics and also within recrystallized micron size grains at the mosaic boundary zones. Nitrogen enriched areas and nitride phase were detected in the inner microstructure of the laser melted samples. The measured tensile strength, yield strength and microhardness were 1214 MPa, 1321 MPa and 450 HV, respectively, which is superior to that of conventional ferritic, austenitic and duplex stainless steels. The Enhanced mechanical properties are due to a number of nano- and microstructure factors such as the nano-sized dislocation loops restricting dislocation movements, different crystalline grain orientation of grains within the mosaics and boundary inclusions and precipitates that inhibit slip/slide effects. Despite of high strength and hardness, the laser melted ferritic steel was very ductile according to stress-strain curves and fracture analysis.

1. Introduction

Duplex stainless steel (DSS) has an approximately equal phase balance of body-centered cubic (BCC) Ferrite and face centered cubic (FCC) Austenite in its microstructure [1]. DSS have gained popularity and have been widely used due to its good mechanical properties and corrosion resistance, which depend on the presence of both the Austenite and Ferrite phases in the microstructure. The Austenite contributes to toughness and resistance against corrosion and the presence of the Ferrite improves strength but decreases crack susceptibility and toughness. DSS has good welding ability and higher strength than any comparable austenitic or ferritic stainless steels. The unique properties depend on the phase balance. DSS is suitable for chemical and petrochemical industry, pulp and paper industry, power generation, marine constructions and offshore structural industries [2–3]. The important phase balance found in the DSS can be disturbed by composition shift and thermal factors such as fabrication cooling rates, which will negatively influence the mechanical properties of DSS. Thus, the processing/fabricating method of DSS is crucial for allowing a correct phase balance and microstructure [4–8]. During welding the melting and solidification destroys the favorable duplex microstructure of DSS because the cooling and heating rates during melting and solidification will change the chemical phase balance [9–11]. The increase in the Ferrite volume fraction will lead to deterioration of toughness of DSS. Moreover, detrimental intermetallic phases such as the brittle sigma phase (σ) could precipitate in the grain boundaries of the DSS microstructure. The sigma phase is precipitated through a eutectoid reaction of the type: $\delta \rightleftharpoons \sigma+\gamma$. This reaction is unbalanced toward the right, *i.e.* in the direction of sigma phase formation [12–13]. It is practically impossible to prevent the formation of sigma phase during solidification cooling, because the DSS compositional range favors its precipitation [14]. The volumetric fraction of sigma phase can be minimized however by increasing the cooling rate during the solidification process. The extremely high and local cooling rates (10^5 to 10^7 KS^{-1}) in laser melting (LM) can maintain the chemical composition of the DSS.

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The aim of this study is to report fabrication of a novel stainless steel with remarkable mechanical properties while maintaining the chemical phase balance and also to investigate the microstructure-property relationship by means of careful electron microscopy studies.

2. Experimental

Nitrogen-gas atomized spherical Duplex steel powder SAF2507 (supplied by Sandvik Osprey Ltd., Neath, UK) with particle size of 32–45 μm was used as starting material. The overall chemical composition of the as-received powder provided by the manufacturer is 25Cr, 7Ni, 4Mo, 0.8Si, 0.3N, 1.2Mn, <0.03 C (all in wt%). Laser melting was performed in Ar atmosphere of 1 bar and the laser chamber had residual oxygen of 0.1 vol%.

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 190 W, scan speed of 750 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 with the angle between each layer 45° as shown in the schematic in Fig. 1.

Test cubic samples and tensile rods were built with dimensions of $10 \times 10 \times 5$ mm and $40 \times 4 \times 1$ mm, respectively. 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.

X-ray diffraction (XRD) pattern was obtained using CuK_α radiation in a Panalytical XPert alpha 1 diffractometer over a 2θ range between 30° and 100°. The JCPDS-cards 31-0619 and 06-0696 was used for identification of Austenite and Ferrite, respectively. The Rietveld method was used to estimate the amount of each crystalline phase.

Macro- and microstructure observations were carried out by an optical microscope (OM, Olympus SZX12, UK) and a scanning electron microscope (SEM, JSM-7000F, JEOL, Tokyo, Japan). Before OM and SEM observations the samples were electro etched in 10% oxalic acid at an operating voltage of 5 V. For deeper microstructural studies 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, JEOL, Tokyo, Japan). 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. Electron backscattered diffraction (EBSD) phase mapping and grain orientations were obtained by TESCAN MIRA 3LMH scanning electron microscope (TESCAN, Brno, Czech Republic) equipped with a HKL Nordlys orientation imaging microscope system (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.

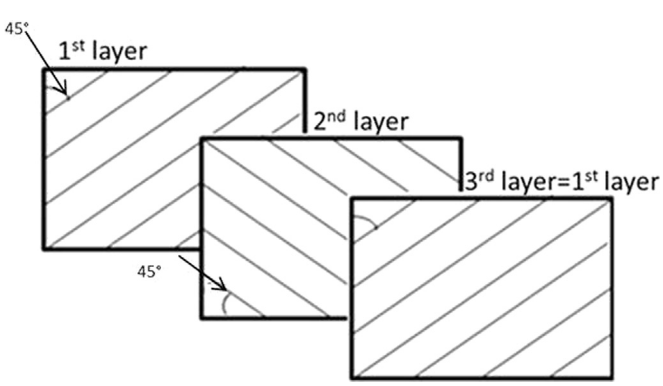

Fig. 1. Schematic of the laser scanning strategy used for making the steel samples.

The Ferrite content of the laser melted samples was measured according to the magnetic induction method by a Feritescope (FMP30, Fischer Gmbh, Germany).

The amount of nitrogen inside the powder and laser melted bulk samples were measured using EXTR Leco TCH600 according to ASTM E1019. The analyzer is designed for wide range measurement of nitrogen content of inorganic materials, ferrous and non-ferrous alloys and refractory materials using inert gas fusion technique.

Tensile strength and yield strength were measured using a standard testing machine (model TIRAtest 2300+upgrade, Schalkau, Germany) with the pneumatic grips and the cross head speed 0.5 mm/min. The values for the mechanical data is an average obtained from 4 identical specimens.

Conventional microhardness has been measured using standard microhardness tester (model Leco LM700AT, Leco Corporation, USA) with the Vickers indenter at the load of 1 kgf and dwell time of 10 s.

3. Results and discussion

3.1. Chemical composition and phase stability of LM steel

The XRD pattern of the starting duplex powder and the laser melted sample is shown in Fig. 2. As seen the powder is almost a single Ferrite phase with small trace of Austenite phase in which the Austenite peaks are present at 2θ of approximately 43, 50 and 74° while Ferrite peaks are present at 44, 64, 81 and 98°. However XRD of laser melted sample shows a fully ferrite with no traces of the crystalline Austenite or any secondary sigma phase. Thus, it can be proposed that processing DSS with laser melting can maintain the chemical phase balance and totally avoid formation of sigma phases.

To approximately estimate the amount of each phase, Rietveld method was used and it calculated ~98% Ferrite and ~2% Austenite. The precursor powder was produced through gas atomization and the high cooling rate during production caused suppression of the Austenite phase. Thus, although the powder chemical composition is corresponding to SAF 2507 duplex stainless steel it contains very little Austenite. Feritescope measurement was also carried out for confirmation of the Rietveld prediction. The result of ferritescope measurement showed about 75–78 vol% ferrite content in the laser melted sample.

The Schaeffler diagram was also used to estimate how much of each phase is expected using Cr_{eq}/Ni_{eq} ratios [15]. The Cr_{eq}/Ni_{eq}