Optics and Laser Technology 108 (2018) 487–495

Contents lists available at ScienceDirect

Optics and Laser Technology

journal homepage: www.elsevier.com/locate/optlastec

Full length article

Sensitisation behaviour of drop-deposited 11% Cr ferritic stainless steel

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ARTICLE INFO

Article history:
Received 3 March 2018
Received in revised form 22 June 2018
Accepted 14 July 2018

Keywords:
Laser additive manufacturing
Drop-deposition
Stainless steel
Sensitisation

ABSTRACT

For low-chromium ferritic stainless steel, a recently developed laser-driven drop-deposition technique enabled the building of three adjacent tracks on a substrate sheet of the same alloy, to study its risk for sensitisation from certain sequences of thermal cycles. The process was recorded by high-speed imaging to understand the drop-deposition mechanisms. Higher beam power resulted in a smoother track. The added layer was fully martensitic, achieving an elevated hardness of 320 HV. For a temperature peak just below austenitisation, the thermal cycle from a subsequent track affected the former track through tempering. Etching revealed a continuous region of ditched grain boundaries around the interface between the melted and heat affected zones. In the melted zone, the network became discontinuous approaching the surface, meaning that the specimen was immune to sensitisation, in contrast to transformation hardening results in the solid state. Additive manufacturing can induce manifold sequences of thermal cycles, but from the here generalized knowledge, strategies against sensitisation can be derived.

1. Introduction

Low-chromium ferritic stainless steel is a promising steel grade but bears the risk of sensitisation when experiencing multiple heating cycles during thermal manufacturing processes. A new technique of laser-driven drop deposition enables the study of this type of steel for additive manufacturing, with respect to sensitisation behaviour of overlapping tracks.

The steel grade EN 1.4003 which is a 11% Cr ferritic stainless steel was investigated. This alloy is commonly used in mildly corrosive environments such as automotive exhaust systems. This steel contains little to no nickel and thus has a lower price than austenitic stainless steels. Ferritic stainless steel grades also have the advantages of higher yield strength, better machinability, and a higher resistance to chloride-induced cracking when compared to austenitic stainless steel. Welding of low-chromium ferritic stainless steels has been studied extensively in recent years due to a development in alloy compositions and increased use in industry. Welding processes are to a certain extent representative for surface treatment because the microstructure formation and the resulting corrosion behaviour can be similar, in the weld metal and in the HAZ, depending particularly on the material, thermal cycle, chemical diffusion and melt flow. Pekkarinen and Kujanpää [1] showed that the microstructure of EN 1.4003 becomes fully martensitic for a range of different laser welding parameters as well as for GTA-welding. When laser welding 12 mm thick 12% Cr stainless steel plates, Taban et al. [2] identified grain coarsening in the high-temperature heat affected zone, HAZ, as a main drawback, since it can reduce the impact toughness. Different filler materials for welding of EN 1.4003 stainless steel were analysed by Anttila et al. [3] The consumable 409LNb led to a dual-phase structure with a ferrite matrix and martensite in between the ferrite grains, while the HAZ was entirely martensitic. The steel grade EN 1.4003 is prone to sensitisation, which was observed as an accompanying effect for welding for certain heating cycles and alloy compositions but never directly investigated for hardening or additive manufacturing. The ASTM A 763-15 standard is applicable to investigate the susceptibility to intergranular attack. According to the standard one or more grains need to be completely surrounded by ditches to be susceptible for intergranular corrosion [4]. For laser welding of EN 1.4003 sheets that were either hardened before or after welding, Dahmen et al. [5] investigated the sensitisation behaviour. Etching of pre-hardened welded sheets showed a ditched structure in the HAZ while the post-hardened samples showed no ditching. The cause for sensitisation is the mechanism of chromium depletion, which occurs under certain conditions when chromium carbides and nitrides, typically $M_{23}C_6$ and Cr_2N, are formed (M are metallic elements where usually Cr is present); these precipitations deplete the adjacent region of chromium, lowering the corrosion resistance of this region. Different measures can impede the formation of chromium precipitates.

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https://doi.org/10.1016/j.optlastec.2018.07.031
0030-3992/

Nomenclature

AM	additive manufacturing	KFF	Kaltenhauser ferrite factor
BCC	body-centred cubic	LAM	laser additive manufacturing
CFD	computational fluid dynamics	PBF	powder bed fusion
CYCLAM	recycling by laser to feed additive manufacturing	P_L	laser power
DED	direct energy deposition	RFC	remote fusion cutting
DOD	drop on demand	TRL	technology readiness level
FCC	face-centred cubic	T_1	critical temperature for precipitation
GMAAM	gas metal arc additive manufacturing	T_A	austenitisation temperature
HAZ	heat affected zone	T_L	liquidus temperature
HSI	high speed imaging		

One route is to keep the carbon and nitrogen content low, while another way is to stabilise the steel with chemical elements like Nb and/or Ti, due to their higher affinity than Cr to form carbides and nitrides, hence maintaining its corrosion resistance. To stabilise ferritic stainless steel, the proportion of Nb and Ti compared to C and N is of major importance. Amuda and Mridha [6] present three different equations relating to balancing the stabilising elements. A review of research on the sensitisation dynamics is given in [6]. If the composition of the alloying elements cannot be controlled, sensitisation must be avoided by controlling the heating cycle.

During welding studies, four different modes of sensitisation have been identified [7]:

> *Mode 1: Sensitisation due to welding incorrectly annealed material*, which occurs when the base material is not annealed and contains untempered martensite before welding.
> *Mode 2: Sensitisation in welds with overlapping heat-affected zones*, which has the same mechanism as Mode 1 but occurs when the untempered martensite is formed by a previous weld pass, usually in the HAZ.
> *Mode 3: Sensitisation due to continuous cooling after welding at low heat input* that occurs in the region close to the fusion line. This mode does not require any previous heat treatment and is critical for materials where the high temperature HAZ is mostly ferritic.
> *Mode 4: Sensitisation when welding with an excessively high heat input* can occur during its slow cooling cycle. Here sensitisation takes place between the high-temperature HAZ and low-temperature HAZ, being the least common type of sensitisation.

Understanding of the behaviour of ferritic stainless steels like EN 1.4003 when applied for additive manufacturing, AM, is of strong interest, particularly with respect to risk of sensitisation because of the superposition of heat cycles resulting from the many generated tracks and layers. AM has seen a growing interest both in industry and research in recent years. AM is based on powder or wire feeding, but the choice of metal grades is very limited, including only a few stainless steel grades. Samarjy and Kaplan [8] recently presented a new technique where material is transferred from a feeding sheet to the workpiece through molten droplets. This technique enables to feed AM with literally any metal grade that is available as solid sheet.

Quasi-steady state laser-induced melting and boiling causes a controlled melt flow down the sheet thickness. The ejected melt generates a drop jet as the tool that feeds additive manufacturing, on a closely located substrate or part. The technique can be carried out by a variety of methods. For the initial feasibility study, the droplets were produced by laser remote fusion cutting, RFC, of the upper feeding sheet and then dropwise deposited on the lower substrate sheet. RFC is deemed more suitable than the more established gas-assisted laser cutting since the melt is then driven by the ablation pressure [9–11] instead of having a high pressure gas jet which can disturb the deposition on the substrate sheet. This drop deposition technique is also capable of efficient direct recycling with a laser beam, of waste or surplus metal to additive manufacturing of a new part, for which it was termed CYCLAM. [12] This LAM-technique turned out to be highly suitable to apply and study 11% Cr ferritic stainless steel EN1.4003 from sheet metal, due to this alloy composition not being available as wire or powder.

The material supply in the drop deposition technique differs from the traditional wire and powder techniques like powder bed fusion, PBF, or direct energy deposition, DED where melting of the material takes place close to the substrate and under influence of the laser beam. Instead, the technique has more similarities with gas metal arc additive manufacturing, GMAAM, where material is deposited dropwise during arc-pulsing. Luo et al. [13] measured the impact energy from the droplets entering the melt pool by acoustic emissions and found that the impact energy decreased with higher pulse frequency. Chen et al. [14] investigated the microstructure and mechanical properties of 316L-samples generated by GMAAM. The tensile properties at room temperature fulfilled industry requirements. Another technique that utilises material deposition through drops is drop-on-demand, DOD, which is mainly used for polymers where drops are deposited through a nozzle [15]. DOD techniques can also be laser-based like laser droplet generation where the end of a feeding wire is melted before it detaches, through gravity, and then lands on the substrate sheet [16,17].

The drop-deposition techniques have several complex fluid flow mechanisms involved. During the here applied technique occurs boiling-driven melt flow, detachment of droplets from the feeding sheet and their incorporation into the track melt pool and substrate, which is accompanied by melt pool flow, boiling recoil pressure from the transmitted laser beam and solidification. The detachment of droplets, based on Plateau-Rayleigh instabilities, has been studied for electrical drop-on-demand generation [18], where droplets of high- and low-conducting liquids were tested and simulated. The simulation showed good agreement with experiments and could be a useful tool to apply for the drop deposition technique studied here. The resulting surface geometry in melt deposition will be dependent on certain processing parameters, particularly feeding rate and beam diameter. Bergemann et al. studied deposition of drops on a layer of the same fluid [19]. In their study they found that the behaviour of small and large droplets was basically the same. Kulju et al. [20] used HSI, computer-vision and CFD simulation to investigate droplet dynamics on hydrophobic and super-hydrophobic surfaces, turning out to be a suitable methodology to validate simulations of droplet dynamics, for different drop oscillation modes.

The surface morphology from a process can be influenced by the shielding gas, as was shown for welding by Krolczyk et al. [21]. The