The effects of heat treatment on microstructure evolution of 17-4 PH single tracks deposited by AM-DED

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Direct Energy Deposition (DED) is an additive manufacturing technique that has received increasing attention from several industrial fields to produce near-net-shape parts, for repairing components or applying hard-facing coatings. In this context, the 17-4 PH stainless steel is an attractive material because it combines good corrosion resistance with high toughness and strength. In the present work, the effects of post-building heat treatment routes applied to 17-4 PH single tracks deposited by DED on an AISI 316L plate were investigated and preliminary results are shown. Optical microscopy was used to study the effects of solubilization and different aging times on the microstructure, focusing on the amount of δ ferrite inside the tracks. Moreover, Vickers microhardness tests were performed to analyze the evolution of hardness across the substrate/track interface.

KEYWORDS: 17-4 PH, DIRECT ENERGY DEPOSITION, HEAT TREATMENT, MICROSTRUCTURE, MICROHARDNESS

INTRODUCTION

Direct Energy Deposition (DED) is one of the most promising additive manufacturing (AM) techniques that has been widely used in the last years for printing complex metallic parts, but also for repairing components or applying hard-facing coatings [1–4]. By means of a focused heat source, typically a laser, the metal is melted simultaneously as it is deposited, and the subsequent rapid solidification promotes peculiar microstructures affecting morphology and distribution of phases with respect to the typical microstructure obtained with casting or other more traditional manufacturing processes. In metal additive manufacturing, the 17-4 PH stainless steel is one of the most used because of its fascinating properties, such as high strength, toughness and corrosion resistance [5–8]. It is well-known that the interesting mix of its properties needs to be generally optimized by means of a precipitation hardening treatment so, also dealing with additive manufacturing techniques, a post-processing procedure is usually necessary in order to improve both the microstructural and mechanical behavior of the steel. Different authors dealt with the improvement of the 17-4 PH mechanical properties by studying the microstructure of samples fabricated via Selective Laser Melting (SLM) or in general

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Powder Bed Fusion (PBF) techniques. Only in a few studies the effect of post-processing heat treatments was investigated, such as in the work of Sun et al. who found that the usual H900 heat treatment performed on 17-4 PH SLMed specimens is able to guarantee values of hardness higher than the ones achieved on 17-4 PH wrought products [9]. Similarly, Cheruvathur et al. highlighted that a homogenization heat treatment carried out at 1150 °C improves the quantity of martensite in 17-4 PH samples produced via AM, reducing the retained austenite with respect to the as-built condition [9]. Several other studies focused on the application of different heat treatment routes on 17-4 PH 3D printed samples correlating their microstructure with the mechanical behavior [10-15]. Nevertheless, to the knowledge of the authors, the analysis of both microstructure and mechanical properties of the 17-4 PH steel processed by DED and thermally treated has not been deeply studied in the literature. Mathoho et al. compared the microstructure and hardness of 17-4 PH DED samples in both as-built and post-process heat-treated conditions [12]. In the present investigation, two different heat treatment routes concerning both solubilization and different aging times are studied to analyze their effect on the microstructure as well as on the hardness of 17-4 PH single tracks deposited by DED onto an AISI 316L stainless steel.

MATERIALS AND METHODS

Three 100 mm long single tracks were deposited over a 120 x 40 x 10 mm AISI 316L substrate (Fig. 1a) via DED by means of a six-axis AMM IRB 4600 robot, just as shown in Fig. 1b. The process parameters used during the deposition are depicted in Tab.1. To perform the analyses presented in this work, three samples from tracks named A and C were drawn at the same distance from the initial point of the tracks in the zone depicted with green diamonds in Fig. 1b; the samples were named A1, A2 and A3 for track A and C1, C2 and C3 for track C (see Fig. 1a). Samples A1 and C1 were used to characterize the material in the as-built condition, while the other samples were subjected to two different heat treatment routes.

The first heat treatment route (HT1), performed on samples A2 and A3, was a modification of the conventional H900 treatment specified for the 17-4 PH steel in the ASTM A693-0; in particular, it involved a solubilization performed at 1040 °C for 30 min, quenching in water and two different aging at 480 °C for 60 min (HT1_60) and 300 min (HT1_300). The second heat treatment route (HT2) was performed on samples C2 and C3, and consisted of a shorter solubilization performed at 1040 °C, quenching in water, and the same aforementioned agings at 480 °C for 60 min (HT2_60) and 300 min (HT2_300). In Tab. 2 are summarized the investigated conditions.

The solubilization heat treatment was performed on a tubular Lenton LTF oven while a Remet mod. E-79N oven was used for aging. During the treatments, a K-type thermocouple was positioned inside the equipment and the temperature evolution was monitored and acquired by an Omega TC-08 data logger with PicoLog 6 software.

Samples in the as-built condition (A1, C1) and after heat treatments (A2, A3, C2, C3) were prepared for microstructural investigation according to standard metallographic procedures. The samples were etched with the Kalling's solution (1.5 g $CuCl_2$, 33 ml HCl, 33 ml ethanol, and 33 ml of water) for 14 s in order to study the microstructural evolution of the 17-4 PH deposited tracks according to the heat treatment parameters via a Leica Dmi8A optical microscope. Moreover, Vickers microhardness profiles were carried out on all the samples across the substrate, the interface and the deposition track by means of a Future-Tech FM1e micro indenter using a 50 kg_f load in accordance with the UNI EN ISO 6507 standard.

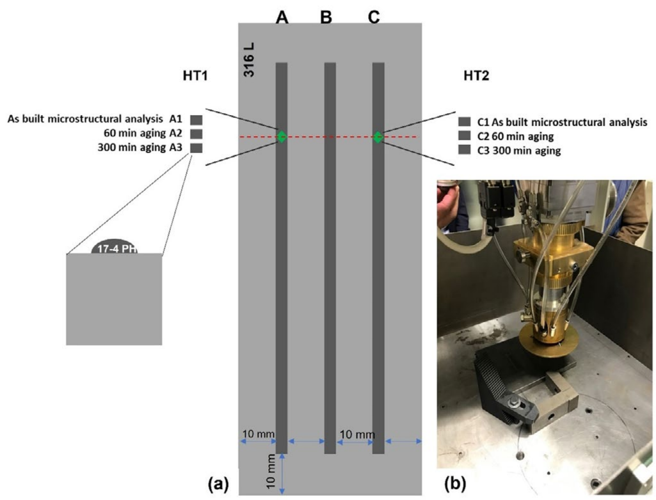

Fig.1 - (a) Scheme of the 17-4 PH depositions tracks, (b) DED equipment / (a) Schema delle tracce in 17-4 PH depositate mediante DED, (b) Attrezzatura DED

Tab.1 - DED process parameters / Parametri di processo DED

Power (p) [W]	Scanning speed (s) [mm/s]	Powder feed rate (fr) [g/min]	Hach spacing (h) [mm]
2160	20	20	1

Tab.2 - Heat treatment routes and samples' codification / Trattamenti termici e codifica dei campioni

Heat treatment	Aging time [min]	
	60	300
HT1	HT1_60 (sample A2)	HT1_300 (sample A3)
HT2	HT2_60 (sample C2)	HT2_300 (sample C3)

RESULTS AND DISCUSSION

In Fig. 2 the typical microstructure of the deposited track is shown, revealing a martensitic matrix with the presence of a lathy δ-ferrite mainly localized at the interface between the deposition clad and the substrate (red blow-up). Islands of interdendritic δ-ferrite were also detected inside the clad, as it can be noted in the light blue blow-up of the same figure. A decrease in δ-ferrite content was observed moving from the interface to the top of the deposition track, in good agreement with some studies [12,16,17] but also in accordance with the thermal gradients and the high cooling rates experienced by the material during the deposition process.

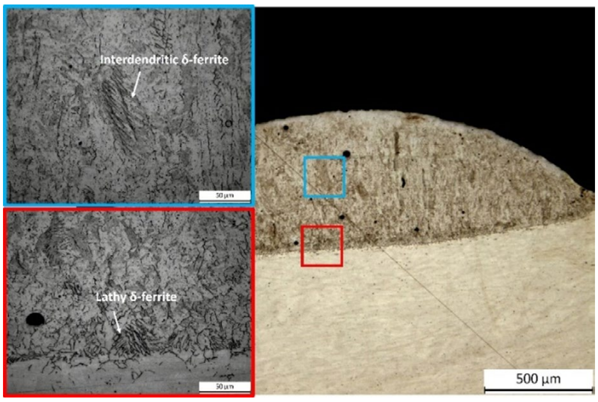

Fig.2 - Microstructure of sample A1 (as-built condition) / Microstruttura del campione A1 (condizione as-built)

Deep microstructural analyses were also performed after the heat treatments. Analyzing the microstructure of samples A2 and A3, some peculiar differences were noted with respect to the as-built condition (see Fig. 3). In both samples, the 30 min of holding time at the austenitization temperature (HT1) was sufficient to guarantee the formation of highly homogenized martensite after quenching. These heat treatment parameters promoted the dissolution of the δ-ferrite. Despite the shorter solubilization time (HT2), no traces of δ-ferrite were evenly detected in samples C2 and C3 (see Fig. 4).

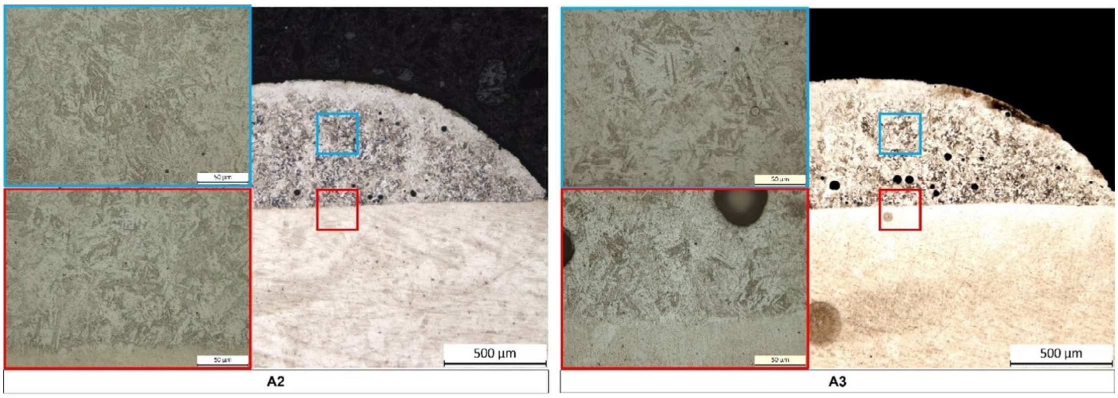

Fig.3 - Microstructure of samples A2 (HT1_60) and A3 (HT1_300) / Microstruttura dei campioni A2 (HT1_60) e A3 (T1_300)

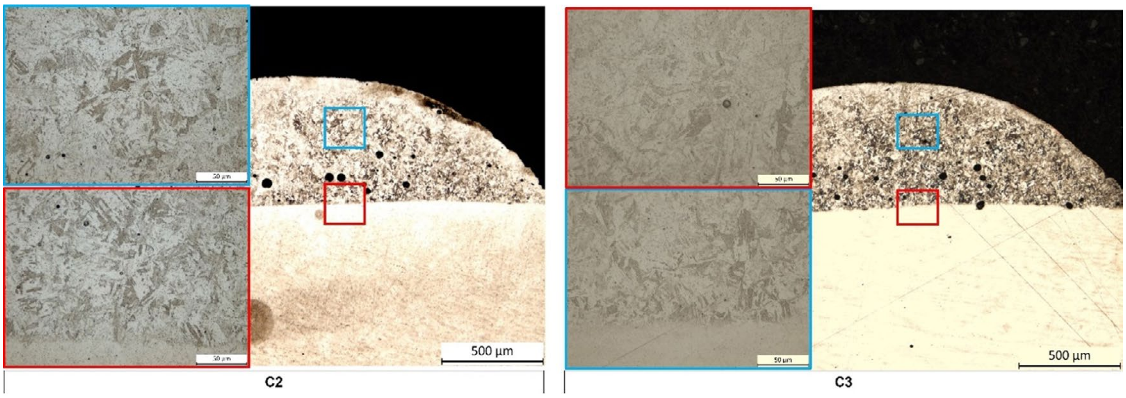

Fig.4 - Microstructure of samples C2 (HT2_60) and C3 (HT2_300) / Microstruttura dei campioni C2 (HT2_60) e C3 (HT2_300)

Microhardness tests were also performed in order to study the efficacy of possible heat treatment routes in enhancing the mechanical properties of the 17-4 PH deposited by DED. A mean hardness of 385 ± 3 HV0.05 was measured for the 17-4 PH steel inside the clad in the as-built condition. In Fig. 5 are depicted the relevant microhardness profiles performed across the substrate/track interface of the heat-treated samples. Considering the mean values of hardness from the bottom to the top of the track, a significant increase in hardness of more than 100 HV0.05 was obtained in samples A2 and A3 with respect to the as-built condition. The duration of austenitization was sufficient to solubilize all the elements useful for the subsequent precipitation hardening that occurred during aging; 60 min of aging (HT1_60) is long enough to maximize the hardness of the material. Conversely, the shorter solubilization applied to samples C2 and C3 was not able to induce the same increase in hardness in 60 min (HT2_60); in this case, the material needs more aging time to gain the maximum increase in hardness (HT2_300).

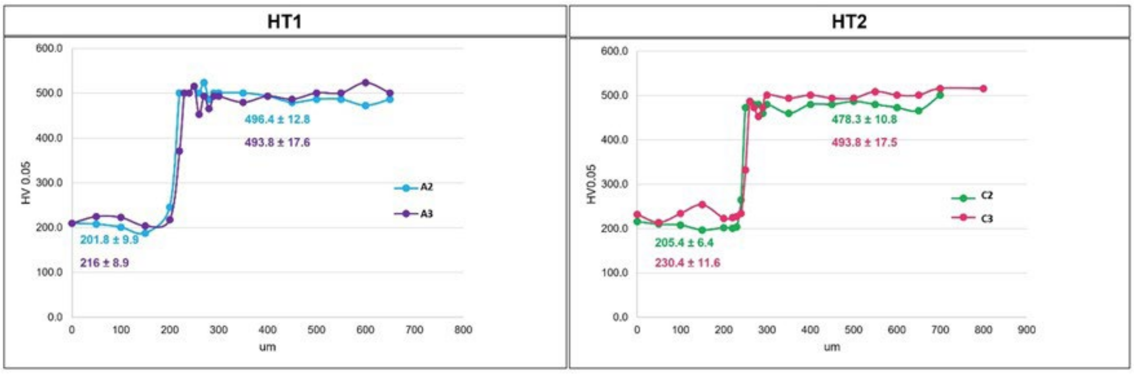

Fig.5 - Representative Vickers microhardness profiles performed on the samples after the investigated heat treatment routes / Profili rappresentativi di microdurezza Vickers eseguiti sui campioni dopo trattamento termico

CONCLUSIONS

In the present work, the effect of different heat treatment routes on microstructure and hardness of 17-4 PH single tracks deposited by DED on an AISI 316L substrate were investigated. The following conclusions can be drawn:

- As-built samples drawn from the 17-4 PH tracks showed a typical martensitic matrix characterized by the presence of intergranular δ-ferrite in the deposited clad. The morphology of δ-ferrite is more lathy across the interface with the substrate. The amount of δ-ferrite decreases from the interface to the top of the track according to the thermal gradient induced by the process.
- The investigated heat treatment routes promoted the dissolution of the δ-ferrite during the austenitization step of the treatment, regardless of the aging time.
- The maximum increase in hardness was obtained with standard solubilization and already after 60 mins of aging (HT1_60). The shorter solubilization route (HT2) is effective in obtaining hardness values comparable with HT1_60 only extending the duration of aging up to 300 mins (HT2_300).

Further investigations need to be performed in order to confirm these preliminary results and other combinations of solubilization and aging times will be explored.

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Studio degli effetti del trattamento termico sulla evoluzione microstrutturale di singole tracce in 17-4 PH depositate mediante tecnologia DED

In molti ambiti industriali, la tecnologia di manifattura additiva Direct Energy Deposition (DED) si sta diffondendo in modo significativo sia per la produzione di componenti near-net-shape sia per effettuare ripazioni o indurimenti superficiali. In questo contesto, l'acciaio inossidabile 17-4 PH risulta un materiale molto interessante sia per l'ottima resistenza alla corrosione sia per le sue elevate proprietà meccaniche. Nella memoria sono presentati i risultati preliminari di uno studio condotto per valutare gli effetti di differenti modalità di trattamento termico sulle proprietà di singole tracce di acciaio 17-4 PH depositate mediante tecnologia DED su di un substrato in AISI 316L. Mediante tecniche microscopia ottica sono stati indagati gli effetti della solubilizzazione e di differenti tempi di invecchiamento sulla microstruttura, con particolare attenzione al contenuto di ferrite δ all'interno delle tracce. Inoltre, prove di microdurezza Vickers hanno consentito di studiare l'evoluzione della durezza attraverso l'interfaccaia substrato/traccia.

PAROLE CHIAVE: ACCIAIO 17-4 PH, DIRECT ENERGY DEPOSITION, TRATTAMENTO TERMICO, MICROSTRUTTURA, MICRODUREZZA

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