A novel approach to direct preparation of complete lath martensite microstructure in tool steel by selective laser melting

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ABSTRACT

A direct preparation of complete lath martensite microstructure (transformation rate $\delta > 99\%$) in tool steel was successfully realized using selective laser melting (SLM) in conjunction with laser remelting (LR) technique. Ultrafine lath martensite with a high percentage of low-angle grain boundaries (LAGBs) (46.12\%) was formed. This unique microstructure contributed to the prominent effect of dislocation rearrangement and entanglement within the substructure of martensite, leading to a significant improvement of mechanical properties. An ultrahigh microhardness of $\approx 765.1$ HV$_{0.3}$ was obtained, which is much higher than the previously reported values of as-built SLM hardened steel such as H13 and maraging steel.

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1. Introduction

Lath martensite, also known as dislocated martensite, is the prime constituent that contributes to high strength in steels. It has immense industrial relevance as it is characteristic for advanced high strength steels (AHSS), e.g. quenching partitioning steel and transformation-induced plasticity steel [1]. Compared with twinned martensite, lath martensite presents an excellent compromise between strength and ductility which shows great potentials to meet the requirements for special engineering components used in tough environments [2]. The traditional method of quenching low-carbon steel to room temperature at a considerably large cooling rate, e.g. oil quenching and water quenching, is used to obtain lath martensite. However, this method is not only low-efficiency and costly, but also difficult to guarantee quality of products, e.g. low hardenability and high quenching failure rate. Moreover, some austenite remains untransformed, referred to as retained austenite, with rapid decrease of temperature to room temperature, which imposes limits on the improvement of hardness and strength of hardened steel. Therefore, a reliable approach to attain a microstructure of complete lath martensite (at least 95\% of the martensitic transformation is completed) within a short period is always of high significance to industrial and academic fields.

Selective laser melting (SLM) is regarded as a promising additive manufacturing technique due to its flexibility in fabricating arbitrary-geometry tools directly from powder material [3]. The extremely rapid melting/cooling stages during SLM process ensure a direct transformation of martensite without any post treatment, as reported by Tan et al. [4]. Nevertheless, the austenite still can not completely transform to martensite during SLM since the martensite tends to retransform to austenite during subsequent melting of an overlying powder layer due to constant heat flow from the molten regions to the building platform [5]. Yan et al. [6] also found that tool steel parts processed by SLM contain a small amount of retained austenite derived from the thermal cycling process. Thus, retained austenite can not be completely eliminated by SLM unless additional technique is applied. In the present study, a direct preparation of complete lath martensite microstructure (transformation rate $\delta > 99\%$) in tool steel was successfully realized by means of “SLM plus laser remelting (LR)” technique. The transformation mechanism of complete martensitic microstructure was discussed. Microstructural characterization and microhardness test were conducted to verify the complete martensitic transformation in tool steel.

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2. Experiment

Spherical, gas atomized 5CrNi4Mo steel powders with a mean particle size of 21.6 μm and high purity of 99.5% were employed as the raw materials. Details SLM procedures have been addressed in [3]. SLM parameters were set at: laser power (P) = 250 W, scan velocity (v) = 1350 mm/s, layer thickness (h) = 30 μm and hatching spacing (s) = 60 μm. In order to obtain a complete martensite microstructure, LR was applied: before adding a new powder layer, each solidified layer was scanned one more time in a P of 125 W, v of 2500 mm/s and s of 50 μm.

Microstructure of the SLM-fabricated part was characterized using a transmission electron microscopy (TEM) (Tecnai G2F30). A NANO SEM 430 (FEI, Hillsboro, Oregon) was employed for electron back-scattered diffraction (EBSD) analysis. The step size was set to 80 nm. The attained data was analyzed using microstructural data analysis software TSL OIM (Ametek Inc, Berwyn, Pennsylvania). The Vickers hardness was measured using a HXS-1000A microhardness tester (AMETEK, China), with a load of 300 g and a dwell time of 20 s. The hardness profiles were obtained from at least 80 indentations made on each surface of sample.

3. Results and discussion

Fig. 1a shows the typical features of complete lath martensite microstructures. A complicated hierarchical architecture was observed, in which a prior austenite grain was divided into packets, and subsequently subdivided into blocks containing multiple laths. The TEM image in Fig. 1b exhibits the morphology of a single martensite lath, in which a heavy dislocation density is well seen. The selected area electron diffraction (SAED) pattern (inset in Fig. 1b) from the (1 1 1) zone axis further demonstrated a typical body-centered cubic (bcc) structure. Since the starting powders used contain a low carbon content (0.5 wt%), it is significantly difficult for undercooled austenite to resolve individual dislocations during the rapid cooling stage, hence forming very tangled arrays. Twining is impeded extensively in this situation and thus only lath martensite is identified in this study. Due to a considerably large cooling rate of melt pool during SLM, the blocks consisting of multiple ultrafine martensite laths showed a significantly refined microstructure with a mean width of around 5 μm. It is of great help to enhance the mechanical property of steel parts since the Hall-Petch relation is proved to be held between the yield strength and block size [7].

Fig. 2 depicts the phase map, orientation map, grain boundary misorientation map and grain size distribution of the SLM-processed steel part by EBSD analysis. The phase distribution has verified that the complete martensite transformation (transformation rate \( \delta = 99.3\% \)) was successfully realized by the “SLM plus LR” technique. Laser remelting of previously solidified layer can lead to a much higher cooling rate because the solid part has a larger thermal conductivity than the powder material. As such, more martensite phase will be generated in the LR-processed parts. From the orientation map, a continuous change of colour, e.g. the red/blue/green regions, was observed. Supposedly, the crystallography orientation of grains in this steel was random. As the orientation map can only identify the high-angle grain boundaries (HAGBs) instead of the substructure of martensite, grain boundary misorientation analysis was conducted and low-angle grain boundaries (LAGBs) were exhibited in Fig. 2b. Since the formation of lath martensites always obeys strict orientation relationship, martensite

![Fig. 1](image_url)

Fig. 1. (a) The typical features of complete lath martensite microstructure; (b) bright-field TEM image of the martensite lath and the corresponding SAED pattern.
laths within a block generally maintain small misorientations. The specific orientation relationship between lath martensite and prior-austenite grains keeps the rotation angle of the substructure boundaries of lath martensite below 5°. Blocks subdivided from packets keep relatively large misorientation in comparison to laths and contribute to the formation of LAGBs (rotation angle ranges from 5° to 15°). Packets formed along different habit planes in an austenite grain typically exhibit remarkable crystallography misorientations. As such, the HAGBs (rotation angle ranges from 15° to 180°) were constituted of two parts: packets boundaries and prior austenites boundaries. The graph of grain size distribution clearly shows that a majority of the grains had a much smaller size below 1 μm, corresponding to the dimension of a single martensite lath. The blocks were identified to be with a size from 1 to 8 μm in which the substructure boundaries of laths exhibit a considerably low rotation angle. It is noted that there are coarse grains (≥8 μm) still existing in the SLM part, as a result of the inevitable thermal cycle from the processing of overlying powder layers.

To better understand the mechanical behavior of tool steel with an unique complete lath martensite microstructure prepared by “SLM plus LR” technique, Vickers hardness measurements were conducted on the top and transverse surfaces of sample and the results are shown in Fig. 3. Both the top and transverse surfaces showed an ultrahigh microhardness ranging from 702.6 HV0.3 to 826.7 HV0.3, verifying the complete martensite transformation. The average microhardness was calculated at about 765.1 HV0.3, which was much higher than the reported value of SLM hardened steel such as maraging steel (645 HV) [8] and H13 (630 HV) [5]. The dramatically increased hardness can be attributed to two factors: complete martensite transformation and increase of sub-grain boundaries. The first factor is understandable as a higher percentage of martensite undoubtedly leads to an enhancement of

Fig. 2. The phase map, orientation map, grain boundary misorientation map and grain size distribution of SLM-processed steel part using EBSD analysis.
hardness. As for the second factor, Wang et al. [9] found that the sub-grain structure is a great contributor to the increase in strength of SLM parts since chemical segregation always prevails both in LAGBs and HAGBs derived from the inherent non-equilibrium solidification process during SLM. From Fig. 2, it is interesting to find that the LAGBs accounted for nearly half of boundary distribution (46.12%), increasing by about 35% compared to the previously published value [10]. The large number of LAGBs significantly impedes the movement of dislocations, thus further elevating the strengthening effect through grain boundaries. The substructure of martensite in the SLM part, which can be regarded as sub-grain microstructure, can bring about prominent effect of dislocation rearrangement and entanglement (Fig. 1), contributing to the ultrahigh microhardness of tool steel prepared by “SLM plus LR” technique.

4. Conclusion

In this study, a direct preparation of complete lath martensite microstructure (transformation rate > 99%) in tool steel was successfully realized using “SLM plus LR” technique. The prepared martensite microstructure was characterized to possess a high percentage of LAGBs (46.12%). The prominent effect of dislocation rearrangement and entanglement within the martensite combined with ultrafine substructure contributed to the enhanced mechanical property of the SLM part. The measured microhardness ($\sim 765.1 \text{ HV}_{0.3}$) was much higher than the reported value of SLM hardened steel such as H13 and maraging steel.

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