INVESTIGATION OF THE MICROSTRUCTURE AND PROPERTIES OF THE AISI H13 TOOL STEEL TI-MODIFIED POWDER DURING LASER CLADDING PROCESS UNDER NITROGEN

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https://doi.org/10.37904/metal.2021.4132

Abstract

Laser cladding (LC) technology allows produce metal-matrix composites from discrete powder materials. Data analysis shows that use of such technology to obtain composite materials with enhanced functional properties is of heightened economical interest. The paper presents the results of research aimed at study the possibility synthesizing dispersion-strengthened steel by hard TiN particles formed during the interaction of elemental Ti powder with N_2 gas. It is added 5 % of VT1-0 titanium powder to the AISI H13 tool steel to form a mechanical mixture. Specimen was synthesized from this mixture on a substrate by laser cladding under nitrogen. Owing to the optical and electron microscopy, X-ray phase analysis as well as microhardness testing it was determined that as a result of synthesis, an alloy of steel with Ti with a lower hardness value is formed compared to the H13 steel deposited in pure form. Uniformly distributed nonmetallic inclusions were found, including titanium nitride in the form of dispersed particles. Thus, it can be concluded that before the titanium powder enters the melt pool, there is not enough temperature and time for diffusion processes with the formation of non-melting TiN particles in the steel matrix. Therefore, this phase is formed from the melt during crystallization. As a result, the high titanium in the form of an alloying element in steel does not lead to the targeted formation of a hardened metal-matrix composite but increases the plastic properties and reduces the quality of track formation during process.

Keywords: Laser cladding, direct laser deposition, alloy synthesis, titanium nitriding, microstructure.

1. INTRODUCTION

Laser cladding (LC), as well as its more modern direction, which implements the principle of additive technologies – direct laser deposition (DLD), has great potential in the field of synthesis of classical materials and new materials. Laser technologies for metal deposition have the advantage of precise local heating with a wide range of adjustable heating zones with a laser spot diameter from 0.5 to 5 mm, laser power from 100 to 10000 W, laser wavelength 500-1200 nm for different types of materials, also modes work with continuous or pulse processing. The main purpose of this technology is the cladding of functional coatings, repair, and additive building of new parts with a strong metal bond of materials and a reduced heating temperature of the substrate. To date, extensive knowledge has been accumulated on the properties of various widely used alloys obtained by these technologies. Scientific papers on the following popular alloys are publicly available: H13 steel, 316L and 304 steel, Ti-6Al-4V titanium alloy, Inconel 625 and 718 nickel alloy [1–4]. The current trends in the development of new materials by this method indicate a high potential for using laser technologies for this purpose. A huge range of rare and less popular alloys that can also be obtained by LC and DLD methods, for example, high-entropy alloys [5], which in terms of hardness, heat resistance, heat resistance, corrosion resistance, wear resistance can compete with traditional special-purpose alloys, shape memory-based alloys, for example, NiTi [6], alloys with dispersion hardening by solid particles, for example, WC for obtaining wear-resistant materials [7].

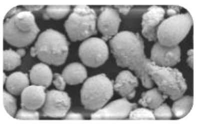
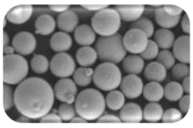
The aim of the work was to create a dispersion-hardened alloy using the example of steel H13 with a stable solid phase from TiN inclusions. Dispersion-hardened materials are those whose resistance to plastic deformation is determined by the deceleration of dislocations on obstacles in the form, as a rule, of nanosized particles. Such structures are obtained in various ways - by separating nanoparticles from a supersaturated solid solution (precipitation-hardening alloys), by powder metallurgy, including mechanical alloying, by internal oxidation and nitriding. The material of the particle is chosen from the most stable compounds — oxides, carbides, nitrides.

The interaction of Ti powder mixed with a die maraging steel H13 in a nitrogen atmosphere in an amount of 5 % in wt is investigated. That is, the effect of nitrogen on the formation of the alloy in the presence of the active element titanium in its pure form is evaluated.

2. METHODOLOGY AND EXPERIMENT

To form the H13/Ti mixture, AISI H13 steel powder produced by TLS Technik in the delivery state and VT-1 0 titanium powder produced by «Normin» were used. The technical characteristics and appearance of the powders are presented in **Table 1**. The raw powder materials were mixed mechanically in a gravity mill for 4 hours. The ratio of powders in the mixture is 95 % H13-5 % Ti (in wt%). The finished mechanical mixture was loaded into the DLD powder feeder. The cladding was carried out on a low-alloy steel substrate with a thickness of 5 mm. Instead of argon, high-purity nitrogen gas was used to supply the powder to the melting zone in the form of a gas-powder mixture and to locally protect the melting zone.

Table 1 Powders for mixing H13/Ti

Powder	Particle size, µm	Particle shape	Chemical composition (w.%)
AISI H13	45 – 90	spherical 	Fe – bal., C – 0.4, Si – 1.0, Mn - 0.48, Cr – 5.3, Mo – 1.3, V – 1.0
Ti (VT-1 0)	45-100	spherical 	Ti – bal., Al – 0.3, Si – 0.01, Fe – 0.15, O – 0.15, N – 0.02, C – 0.021

The laboratory setup for laser cladding, which was used for the experiment, consists of the following components: ytterbium fiber laser iPG LS-3, powder feeder PF 2/2, optical system (cladding head) KUKA MWO-1 and Fanuc M20i robot.

LC was used to build thick-walled samples with a height of 10 mm from a powder mixture of H13-5Ti. Two laser powers were used in the experiment: a reduced power of 1100 W and an increased power of 1400 W. All other technological parameters were fixed, and they are indicated in **Table 2**. The samples were cut, and metallographic sections were made from them in two directions: parallel (XY) and perpendicular (XZ) to the growing direction. Chemical etching of metallographic sections was carried out in a solution of 10 ml HNO_3, 30 ml HCl and 40 ml H_2O. Photos of the microstructure were obtained using a Leica optical microscope. Chemical analysis was carried out on SEM Tescan Mira 3 electron microscope with EDS attachment. X-ray phase analysis was carried out on Bruker D8 Advance diffractometer. Quantitative phase analysis was performed using TOPAS5 software.

Table 2 DLD parameters for growing samples from elementary powders from H13-5Ti mixture in nitrogen atmosphere.

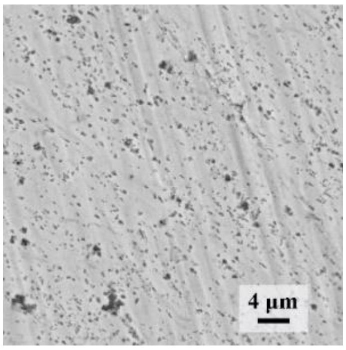
Cladding parameter	Parameter value
Speed of movement	10 mm/s
Powder feed	15 g/min
Shielding gas-nitrogen	15 l/min
Transportation gas-nitrogen	6 l/min
Laser power	1100 and 1400 W
Lifting height	0.6 mm
Offset	50 %
Laser spot size	3 mm

3. RESULT AND DISCUSSION

As a result of the experiment, two thin-walled samples were obtained from a mixture of H13-5Ti (**Figure 1**).

Figure 1 Thin-walled samples grown from a powder mixture of H13-5Ti in a nitrogen atmosphere at a laser power of 1100 W (front sample), of 1400 W (back sample).

SEM photos of the non-etched structure showed the presence of inclusions evenly distributed over the entire volume of the material. The inclusions in the sample grown at a power of 1100 W are evenly distributed in the matrix of the base material and their size does not exceed 1 µm. In the sample grown at a power of 1400 W, there are larger phases of 1-2 microns. The image of the microstructure is shown in **Figure 2**. Some inclusions have a square shape characteristic of TiN nitrides, and the formation of TiC_xN_y carbonitrides is also possible under experimental conditions, but only titanium nitrides were determined by the XRD result. Authors of the work [8] report that micron-sized TiN emissions contribute to the mechanism of steel failure by acting as sites of voids or cracks. However, in this case, their role in the destruction mechanism was reported only for micro-sized TiN secretions [9]. It was reported that cubic or rectangular secretions with sizes from 3 to 90 nm are more effective than spherical secretions in terms of grain growth retardation and increased strength characteristics. Therefore, it can be concluded that the target state of titanium nitride inclusions in steel should be nanoscale particles.

a) 

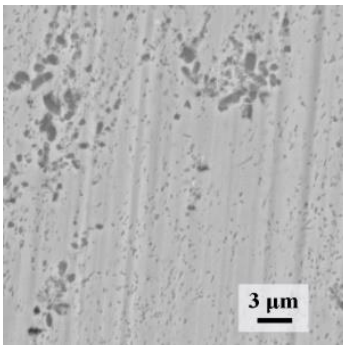
b) 

Figure 2 SEM images of a thin-walled sample in the XZ direction, where a is a sample at a laser power of 1100 W, b is a sample at a laser power of 1400 W

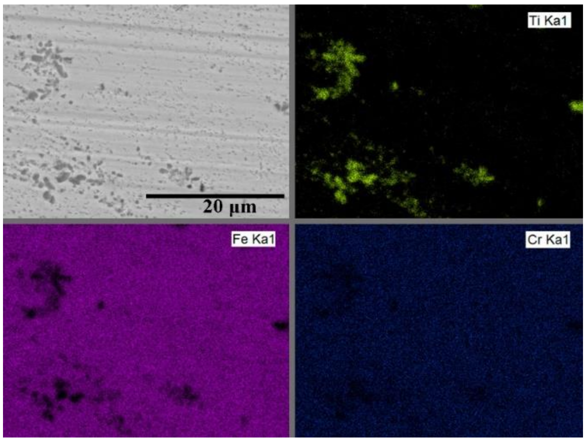


Figure 3 Map of the distribution of elements in the synthesized sample

The distribution of chemical elements in the sample was obtained by the EDS method. **Figure 3** shows that the main component of the inclusions is titanium. The total chemical composition of the material is shown in **Table 3**.

Table 3 Chemical composition of samples obtained by laser cladding from a mixture of H13-5Ti

Element	Si K	Ti K	V K	Cr K	Fe K	Mo L
Weight %	1.08	4.88	1.11	5.42	85.70	1.82
Atomic %	2.11	5.60	1.19	5.73	84.33	1.04

The microstructure of the etched samples, which is shown in **Figure 4**, has no significant differences when changing the laser power or the direction of the study. The structure in the XY and XZ directions also does not differ, which is an interesting result, since the typical structure of the sample obtained by the PLV method has a pronounced texture corresponding to the direction of grain growth towards the heating source, usually at the Z coordinate (up).

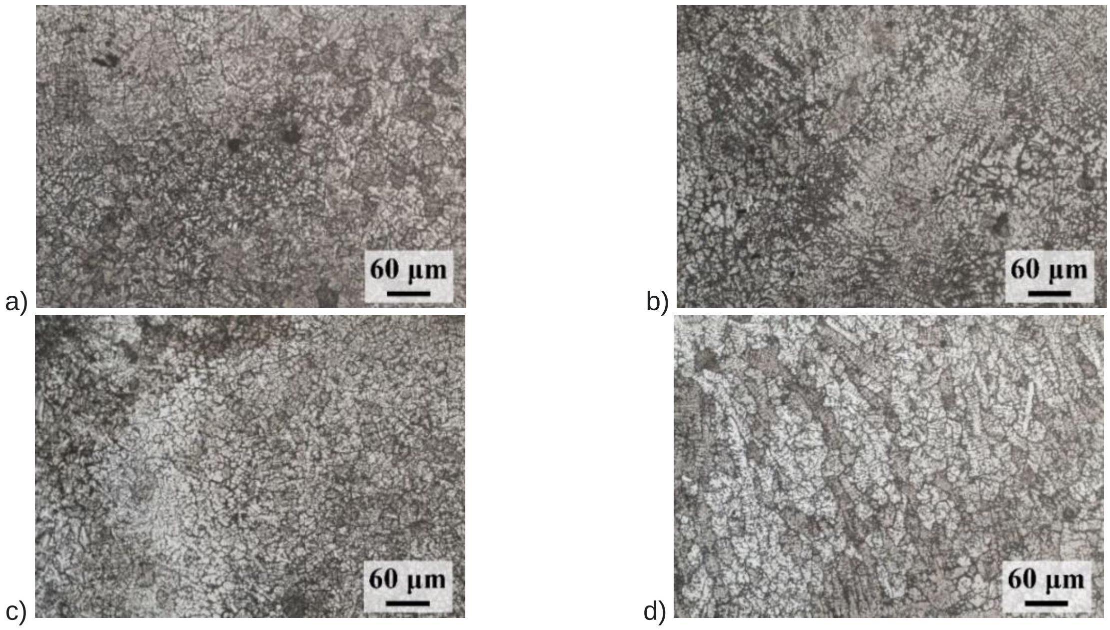
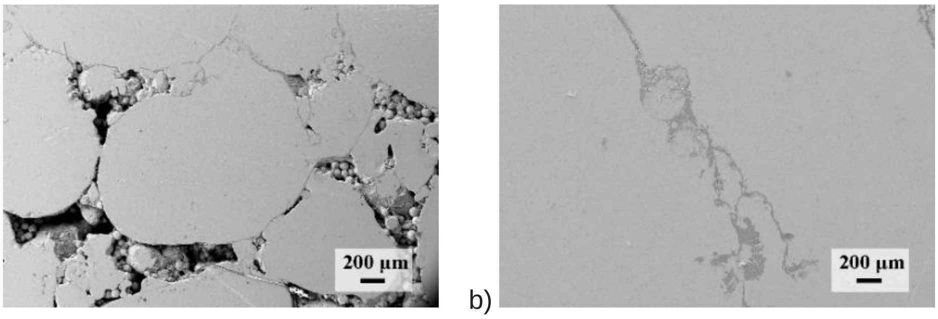


Figure 4 Microstructure of thin-walled samples in an optical microscope at x200 magnification, where a – 1100 W, XY direction, b – 1100 W, XZ direction, c – 1400 W, XY direction, d – 1400 W, XZ direction

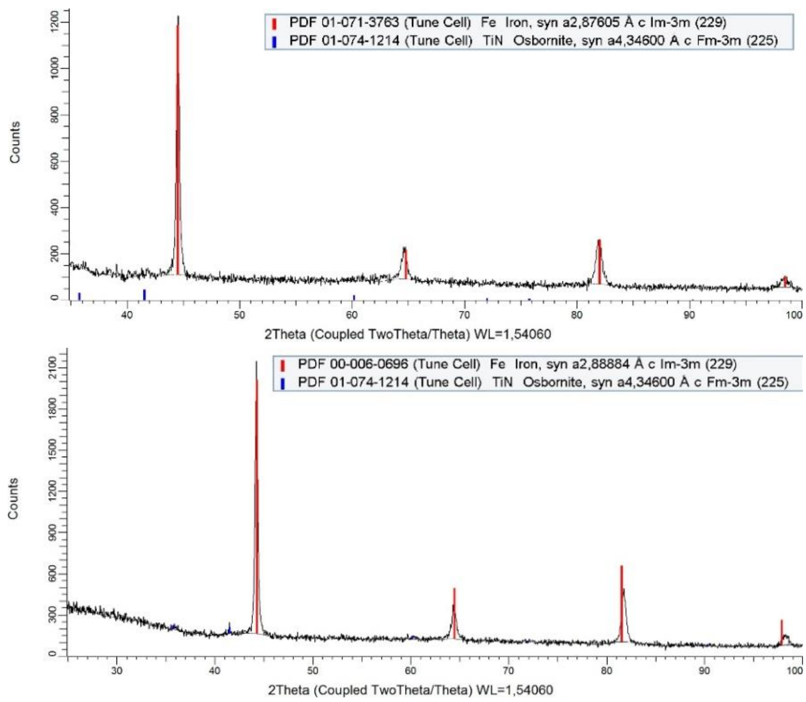
The microstructure of the sample consists of equiaxed dendrites of the first and second order without a pronounced orientation of the growth direction. There are also areas with a grainy structure in the photographs. The average size of the structural components for a sample grown at a laser power of 1100 W is 5 - 10 µm; with an increase in the laser power to 1400 W, the size of the structural components increased to 30 µm. This type of mixed structure and its size is typical of H13 steel obtained by direct laser deposition [10]. Often the structure of this steel obtained by the DLD method has the same (but several components) structure within a single track [1]. Additional mechanical alloying of titanium powder with elemental elements significantly reduced the homogeneity of the structure of steel H13 within each individual track due to high cooling rates during additive building. This effect does not allow the full extent of diffusion mixing processes. For the same reason, there is no clear boundary between each single track.



a) b)

Figure 5 Emerging defects during thin-walled sample building: pores with unmelted powder, and long-range boundary inclusions

The addition of titanium powder to steel H13, as well as a nitrogen atmosphere, significantly reduced the quality of multilayer samples under the same technological conditions that are used for this steel. Defects uncharacteristic for the H13 alloy were found - unmelted powder, pores, and boundary inclusions of a large extent (**Figure 5**). Since LC occurred only with local protection of the melting zone, titanium could form phases with oxygen in the air. To make sure that the nitride inclusions were formed, an XRD analysis was performed (**Figure 6**), since the EDS analysis cannot determine the nitrogen content. The analysis showed that in addition to the main phase of α-Fe, there is a small reflex in the region of 410, which is probably associated with the presence of a small amount of TiN with the HCC structure. The TiN phase was detected in the sample obtained at a power of 1400 W, and this phase was not observed on the diffractogram at 1100 W, probably due to its low content.



a)
b)

Figure 6 XRD of thick-walled samples, a-1100 W, b-1400 W

Typical hardness of H13 steel obtained by LC or DLD for single tracks is 400-650 HV [11], 500-800 HV [Wolosz2020] and for multi-layer coatings 500-650 HV [12]. Since the introduction of a large amount of titanium in carbon alloy steel reduces the hardness and strength characteristics. Since, in addition to the formation of nitrides, titanium binds carbon into resistant carbides, which do not participate in the processes of dispersion hardening.

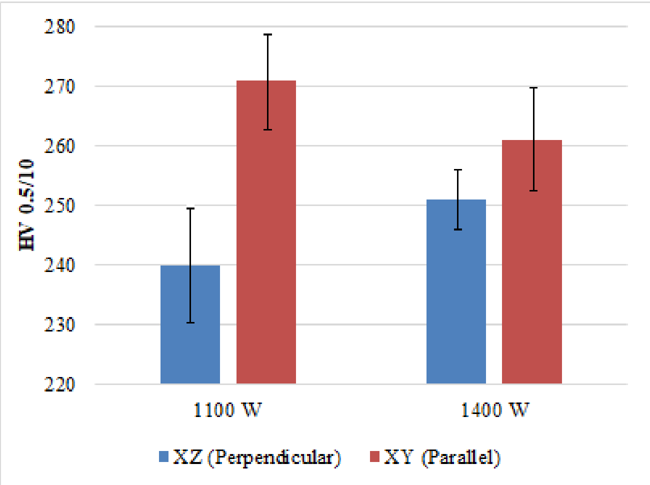


Figure 7 Influence of laser power on specimen hardness in two directions

Figure 7 shows the results of the hardness values in the XZ and XY directions, which indicate a significant decrease in this characteristic compared to a material without the addition of titanium. The change in the laser power mainly affected the difference in hardness in two different directions. With an increase in the laser power, the cooling time of the deposited track correspondingly increases, which reduces thermal stresses inside the material, which affect the hardness.

4. CONCLUSION

The main result of the work is that during the synthesis of an alloy from a mechanical mixture of steel H13 and titanium powder in a nitrogen atmosphere, titanium nitrides are formed. TiN inclusions are formed from the liquid phase after the powder mixture is remelted in a molten bath and titanium is bonded with nitrogen at a high temperature. The size of the inclusions, predominantly located along the grain boundaries in the steel matrix, is 1–2 μm. Mechanical properties, namely the hardness of the samples, significantly decreased and amounts to 240–280 HV, depending on the direction of research and laser power. The results of the work show the possibility of practical application of the nitriding method in the process of laser cladding and direct laser growth with the formation of dispersion-hardening particles.

ACKNOWLEDGEMENTS

We acknowledge the support from Russian Science Foundation grant (project №. 19-79-30002).

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