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Microstructural characterization of laser cladded AISI 316 stainless steel on a carbon steel substrate

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ABSTRACT

Laser engineering net shaping (LENS) is a method of depositing metals into fully shaped parts or for the production of clad layers of noble or wear resistant metals on construction grade steels. In the current work stainless steel was deposited using different combinations of processing parameters such as speed, layer thickness and laser power. The resulting microstructures within the clad layers were then characterized using different etching techniques.
Microstructures formed during the SLM process are comprised of columnar grains with a cellular, cellular/dendritic substructure. The exact shape of these grains is difficult to observe with the use of common etching techniques, this is especially true when considering thin cladded layers, with dissimilar etching behavior. For this purpose we compared a variety of different etchants, which attack the ferrite phase or produce a tint effect. Special attention was giving to the delineation of the columnar grains, which under certain processing parameters can exceed the thickness of individual deposited layers.

Key words: *LENS, stainless steel; microstructural characterization; tint etching, solidification microstructure*

1. INTRODUCTION

Direct laser melting is an attractive group of technologies for the production of near net shaped products, production of advanced materials from dissimilar elements [1] and surface cladding. In the studied example AISI 316 stainless steel powder was deposited on the surface of a low alloy construction grade steel by means of a Selective laser melting (SLM) process, more specifically Laser engineering net shaping (LENS). Laser clad layers are known to develop a superior metallurgical bond when compared to conventional welding, while at the same maintaining a low dilution, thereby resulting in a higher effective thickness, as the second layer already exhibits the desired corrosion resistance.
For a detailed interpretation of the microstructure certain features have to be revealed with the use of proper etchants. The SLM layer solidifies into columnar grains oriented in the direction of heat flow, the substructure of these grains can be either cellular or cellular/dendritic depending on the cooling rate [2]. These features determine the toughness and adhesion of the cladded layer and to some extend even its corrosion resistance.
Etching of dissimilar materials often presents a certain challenge if highly aggressive etchants are required for revealing the microstructure. In the case of laser cladding the height of the deposited layer varies with processing parameters, but generally they are too thin to make their separation a viable option, and the observation of the fusion zone proves to be particularly difficult. For these reasons the substrate must be either protected or preferably an etchant used which doesn't affect it. Different etchants were tested based on this criteria, but generally no one etchant has yielded satisfactory results in revealing all

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microstructural features that are thought to be important. These are the size of the columnar grains, the cellular substructure, fusion lines and the delta ferrite phase.
A modified Beraha 10-3 solution yields the best results in terms of phase, and cell contrast while selenic acid gives a uniform response at low magnifications. The columnar grain size is then observed based on the change in color contrast. The aim of this paper is to determine the optimal approach to the characterization of SLM cladded layers by means of light microscopy and to outline which microstructural features can be characterized using different etchants.

2. MATERIALS AND METHODS

A carbon steel substrate was laser cladded using LENS (Laser engineering net shaping) technology, the processing parameters were discussed elsewhere. The samples were then cut with EDM (electrical discharge machining) and ground using silicon carbide paper up to P600, followed by polishing using 9, 3 and 1 micron diamond suspensions. Prior to etching the less noble substrate had to be protected in certain cases otherwise contaminating the etching solution. The SLM deposited layer was then investigated with light microscopy. The etchants used were 4 % picral, Ralph's reagent which is comprised of 50 ml H2O-50 ml Ethanol-50 mL Methanol-50 mL HCl-1g CuCl2-3.5g FeCl3-2.5 ml HNO3, 10 % aqueous sodium metabisulfite, beraha 10-3 and selenic acid. Beraha 10-3 and 10 % aqueous sodium metabisulfite were modified with a varying amount of HCl, ranging from 15-25 %. 4 % picral and Ralph's reagent are conventional etchants attacking the ferrite phase.
The basis of tint etching using Beraha 10-3 and 10 % aqueous sodium metabisulfite is that the sulfite salt decomposes into S02, H2S, and H2 when it comes in contact with the metal surface. The surface of a stainless steel, becomes depasivised by S02, while the H2S provides sulfur ions, which combine with metallic ions, to produce the sulfide staining film. During etching selenic acid is reduced to elemental selenium and is deposited on the cathodic areas in the microstructure, producing colors that vary with the thickness of the film on the constituents. The alcohol suppresses ionization and polarization at the microelectrodes, permitting preferential deposition on the cathode areas [3]. The phase contrast arises from the misfit of lattice parameters and is therefore most pronounced when the samples are lightly etched and fades with prolonged etching time. A different approach is by applying an tint etch based on selenic acid, whereby elemental selenium is deposited on the metal surface providing an orientation contrast due to varying thickness [4].

3. RESULTS AND DISCUSSION

The general microstructural features can be revealed by etching with 4 % picral [5], this etchant attacks the delta ferrite phase and delineated the cell boundaries, as can be seen in Fig.1, the cell size and preferred orientation are clearly visible, but only little information can be obtained concerning the blocks. In order to characterize the later, and obtain a measure of the effective grain size we applied the commonly used tint etchants, which were modified with varying concentration of HCl. Tint etchants are employed when characterizing materials that are difficult to etch, but in the case of stainless steels an addition of HCL is necessary for the onset of deposition.

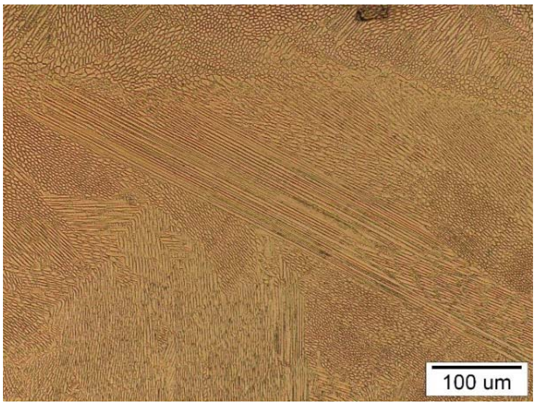

Fig. 1 *Microstructure of sample with visible reorientation of cell-blocks (etched with 4 % Picral).*

Aqueous sodium metabisulfite reveals the orientation of the grains as shown in Fig. 2.

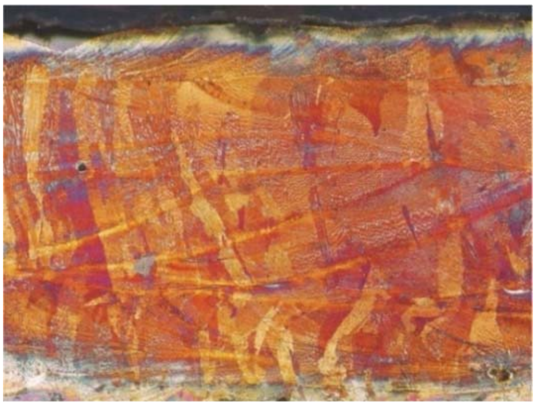

Fig. 2 *Microstructure: a) Cross-section of SLM deposited stainless steel layer*

As well as the ferrite at the cell boundaries and the austenitic interior of the cells as can be seen in Fig. 3, thereby suggesting that the ferrite formed with a eutectoid reaction [6]. Chemical segregations within the heat affected zone are also visible.
In the case of Beraha 10-3 an more pronounced color contrast arises from the different orientations of the columnar grains as shown in Fig. 4a, a strong contrast is attainable by using a low addition of HCl of about 5 % and applying prolonged etching times of up to several hours, in this way the etchant also provides a clear phase contrast at higher magnification as can be seen in Fig. 4b. The observable degree of randomness is quite high, but a certain tendency towards a radial distribution along the weld pool circumference is nevertheless present. This

gives us some indication about the cooling rate. In both cases a uniform etching response was difficult to achieve throughout the whole thickness of the SLM layer.

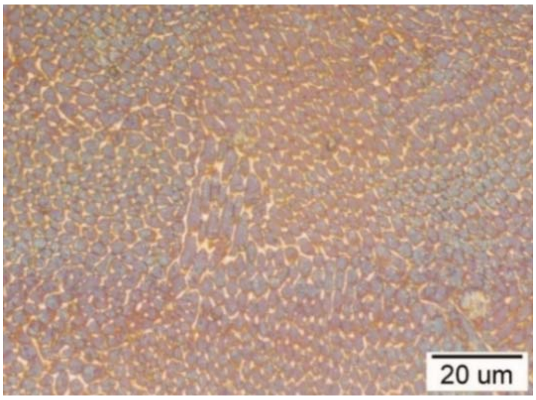

Fig. 3 *Visible phase contrast and cell morphology; white dots are etch artifacts (Etched with 10 % Sodium metabisulfite).*

a)

b)

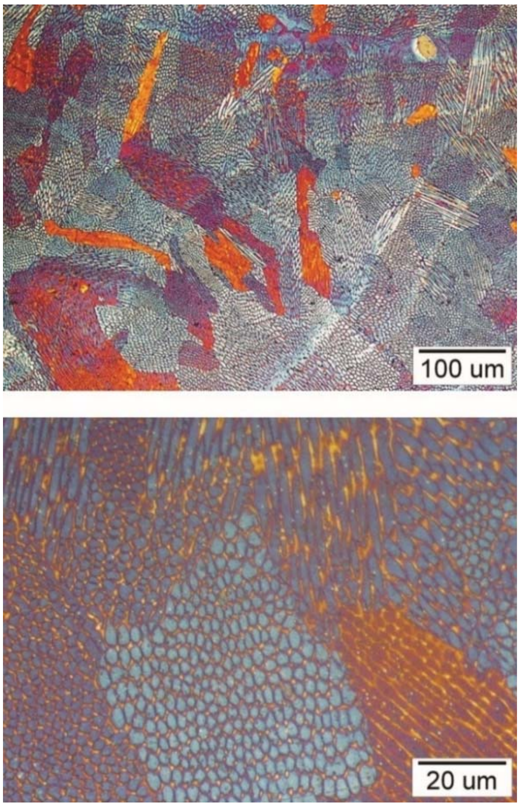

Fig. 4 *Microstructure: a) Cross-section of SLM deposited stainless steel layer; b) Cellular solidification morphology; small white dots are etch artifacts (Etched with Beraha 10-3 with 15 % HCl).*

To obtain a uniform view of the whole of the deposited layer, etchants based on selenic acid we applied, a standard aqueous solution of 1 % and a higher concentration of 3 % were used. In both cases the etching response was uniform across the whole deposited layer and had little to no interaction with the steel substrate. The standard solution delineates the cellular structure, and segregation bands as shown in Fig. 5. However at higher magnifications the cellular structure becomes somewhat less clear using this type of etchant.

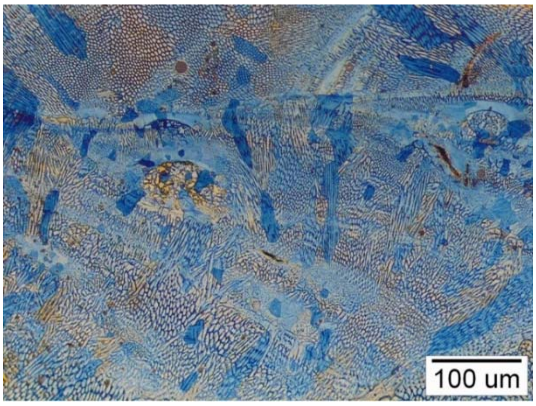

Fig. 5 *Normal cross section of the cladded layer, (etched with 1 % selenic acid solution).*

Higher concentrations of selenic acid don't reveal the cellular substructure as the deposited layer grows thick to rapidly, in this way only the blocks as a whole are delineated as shown in Fig. 6. The effect is different as opposed to using longer etching times as the layers grow uneven due to a preferred orientation of the selenic crystals, resulting in a surface relief which renders the images blurry. A more highly concentrated selenic etch can be used to clearly identify cellular growth which is sometimes confused with an equiaxed transition depending on the observed cross-section [7]. The results obtained using selenic acid are quick and easily repeatable providing the concentration remains stable, the later however diminishes very rapidly as compared to other etchants.

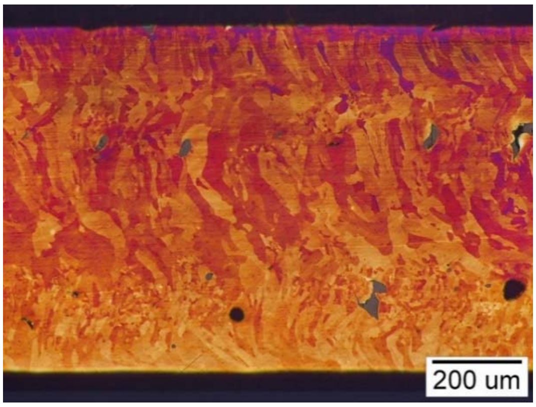

Fig. 6 *Microstructure of sample (etched with 3 % selenic acid solution).*

As there are but few obstacles for the propagation of a crack along the interphase ferrite/austenite boundary, the effective grain size corresponds to the size of the columnar grains. The approach of determining the effective grain size is somewhat in contrast with the findings of other authors that report very fine grained microstructures [8]. However it seems justified from a standpoint of fracture mechanics since the cellular substructure offers little obstacles for the propagation of a crack along the cell boundary. The cells show a slight tendency toward coalescing during the reheating/remelting step. This also influences the morphology of the intercellular ferrite which instead of being distributed preferably homogenously at the austenite cell borders [9], coalesces into fibers, to minimize the surface energy. Because during the laser deposition the preceding layer becomes remelted as well, certain blocks continue to grow through the individual deposited layers by means of epitaxial nucleation. This effectively results in a coarse columnar grain structure. Its occurrence is largely dependent on the processing parameters, and becomes more pronounced with higher speeds and energy inputs, incidentally this is within the processing window that results in a fully dense layer. Directionally solidified stainless steels have been shown to exhibit certain advantages in terms of higher resistance to hydrogen embrittlement [10].

This etchant can also be successfully applied when observing stainless steel welds that have comparable cooling rates as shown in Fig. 7a. However when etching regular welds with selenic acid some addition of HCl in the order of a few percent is required. In order to assess the accuracy of selenic acid a comparison was made with Ralph's reagent and is commonly considered as the etchant of choice for stainless steels containing precipitates as can be seen in Fig. 7b, but the etching response of cladded layers is not appreciably different from the 4 % picral etch, which is due to the pronounced chemical heterogeneity of the clad microstructure.

a)

b)

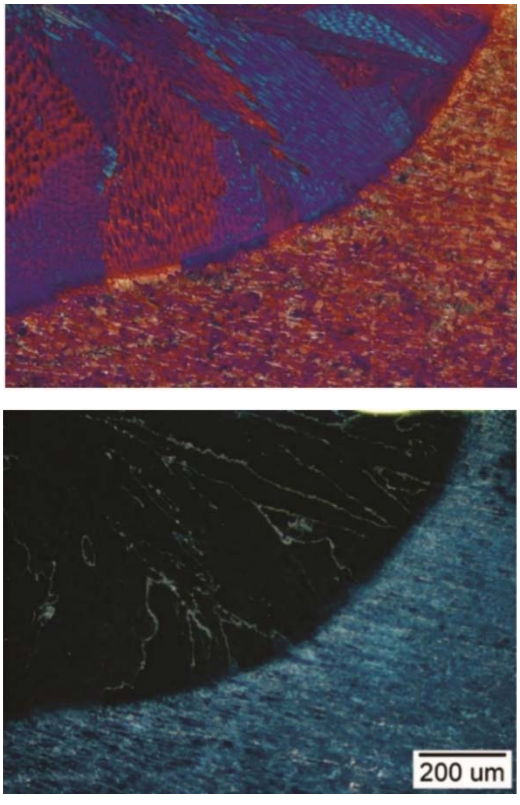

Fig. 7 *Correspondence between tint etching with selenic acid (a) and regular etching using Ralph's reagent (Viewed in dark field mode) (b).*

4. CONCLUSIONS

Metallographic analysis of the tint etched samples has shown that the solidification structure of the SLM deposited layer consists of fine cells of austenite with ferrite present at the cell boundaries. Tint etching has shown to be an effective method of delineating them based on the orientation dependent color contrast. It is observable that the columnar grains begin to grow in the direction of the heat flux, and later continue along preferred crystallographic orientations thereby obtaining a curved shape during growth.

All the etchants used revealed the phase contrast, but the blocks and therefore consequently the effective grain size were only clearly observable with Beraha 10-3 and selenic acid and to some extend using 10 % aqueous sodium metabisulfite. In general etching with selenic acid has yielded the best results in revealing the general microstructure across the entire cladded layer uniformly, fast and with easily repeatable results, as long as the concentration of selenic acid in the solution is maintained. This procedure could prove advantageous in the characterization and production of heat resistant SLM manufactured parts, assuming the cellular structure would annealed out successfully.

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REFERENCES

[1] Articek, U., Milfelner, M., Anzel, I. (2013). Synthesis of functionally graded material H13/Cu by LENS technology. *Advances in Production Engineering & Management*, 8 (3), 169–176.

[2] Fearon, E., Watkins, K. G. (2004). Optimisation of layer height control in direct laser deposition. *23rd Int. Congr. Appl. Laser Electro-Optics (ICALEO 2004)*, 1708, 2004.

[3] Vander Voort, G. F. (1985). *Metallography: Principles and practice*. ASM International.

[4] Vander Voort, G. F., Lucas, G. M., Manilova, E. P. (2004). Metallography and Microstructures of Stainless steels and Maraging steels. *ASM Handbook*, 9, 670–700.

[5] Bramfitt, B. L., Benscoter, A. O. (2003). *Metallographer's guide practices and procedures for irons and steels*. ASM International.

[6] Brooks, J. A., Williams, J. C., Thompson, A. W. (1983). Stem Analysis of Primary Austenite Solidified Stainless Steel Welds. *Metallurgical Transactions A.*, 14 (1), 23–31.

[7] Xu, P., Lin, C. X., Zhou, C. Y., Yi, X. P. (2014). Wear and corrosion resistance of laser cladding AISI 304 stainless steel/Al2O3 composite coatings. *Surface and Coatings Technology*, 238, 9–14.

[8] Li, R., Shi, Y., Wang, L., Liu, J., Wang, Z. (2011). The key metallurgical features of selective laser melting of stainless steel powder for building metallic part. *Powder Metallurgy and Metal Ceramics*, 50 (3), 141–150.

[9] Elmer, J. W., Allen, S. M., Eagar, T. W. (1989). Microstructural development during solidification of stainless steel alloys. *Metallurgical and Materials Transactions A*, 20 (10), 2117–2131.

[10] Liu, J., Li, Y. Y., Hu, Z. Q. (1989). Effect of directional solidification on resistance to hydrogen embrittlement of a stainless steel. *Materials Science and Engineering: A*, 117, 221–226.