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Coating by laser surface treatment (*)

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Abstract. — The application of optical engineering in industrial processing is gaining greater acceptance. The range of surface treatment processes available goes all the way from transformation hardening, annealing, shock hardening and bending (as processes which do not involve melting) to processes which involve melting such as surface melting, surface alloying, surface cladding and those processes which involve some form of photochemistry such as laser chemical vapour deposition, laser physical vapour deposition and stereolithography. One of these processes, laser cladding, has been recently extended at University of Liverpool to provide a technique of rapid alloy scanning whereby variable composition laser clad tracks can be laid down by the laser thus giving the whole range of compositions in the alloy system in a single sample. Evaluation of specimens of this type can be carried out by a range of micro testing techniques including corrosion testing procedures. Laser surface treatment processes available for the production of corrosion resistant surfaces are reviewed. These include surface melting, surface alloying and surface cladding procedures. Consideration is then given to the new variable composition laser melt track technique with particular reference to the rapid prototyping of corrosion resistant alloys.

1. Introduction.

The principal laser techniques employed for the production of corrosion resistant metallic surfaces are laser surface melting (LSM), laser surface alloying (LSA) and laser cladding. Following a summary of the overall advantages and disadvantages of these processes, a brief summary of work on each of these techniques for the enhancement of corrosion properties in the case of steel is presented. (The work that has been carried out on cast iron, aluminium or titanium alloys is not considered here).

1.1 ADVANTAGES AND LIMITATIONS OF LASER PROCESSED SURFACES FOR CORROSION RESISTANCE APPLICATIONS. — The highly localized interaction between lasers and materials makes laser processing an attractive alternative in the realisation of coating strategy for engineering materials. This is particularly the case for the production of corrosion resistant surfaces where the following advantages compared with more conventional techniques can be identified:

i) A dense surface layer with no porosity is produced (cf. metal spraying or electrodeposition techniques where porosity is often a feature);

ii) Integrity of bonding between the surface layer is complete and hence is superior to coating techniques such as spraying, electrodeposition or roll bonding. This integrity of

(*) Keynote lecture.
bonding is particularly important in corrosion resistance applications since failure of the sub-
strate/coating interface is often a critical limitation of conventional coated materials;

iii) Since the depth of penetration can be precisely controlled and the heating effect is
strongly localized, there is no deleterious effect on bulk mechanical properties such as can
occur in surface diffusion techniques involving prolonged heating;

iv) High vacuum conditions are not required since oxidation effects can be simply avoided
by the use of an inert gas shroud (cf. electron beam and physical vapour deposition tech-
niques where high vacuum conditions are required). This implies substantial manufacturing
flexibility for laser processes;

v) There is no fundamental restriction on component shape (cf. roll bonding which is
predominantly applicable to sheet);

vi) Surface microstructures which are unobtainable by any of the main coating or surface
treatment technologies can be produced. Novel microstructures such as microcrystalline or
amorphous surfaces have outstanding relevance for enhancement of corrosion resistance.
Production of surfaces of this type via laser techniques has the advantage over other rapid
solidification techniques in that in principle the whole component surface can be treated in
this way without affecting component integrity;

vii) Limited areas of a component surface can be processed without affecting surrounding
areas and hence these processes are applicable to the limited area processing of critical regions
of a component for corrosion resistance applications.

Despite these advantages, there has been a much larger research effort to exploit the en-
hanced hardness and wear resistance capabilities of laser treated surfaces than to exploit
enhanced corrosion resistance. This can be explained in terms of the limitation in the area
coverage achieved in a single pass in currently available laser processes. Large area cover-
age is dependent on the overlapping of successive tracks of typical width 3-5 mm or less and
hence restrictions are placed on the degree of compositional and microstructural uniformity
that can be achieved on a component surface. While less important in hardness and wear re-
sistance applications, these factors are of vital importance in corrosion resistance applications.
Advances in laser process techniques aimed at increasing the area processed in a single pass
are highly desirable from this point of view. Limited area processing for corrosion resistance
applications would not be subject to this restriction.

In the following sections, unless otherwise stated, the processing referred to was carried
out using a CO$_2$ CW laser.

1.2 LASER SURFACE MELTING (LSM). — In this technique [1, 2] (also known as laser surface
remelting or laser glazing) melting is induced in the near surface by relatively high intensity,
short duration laser interaction (eg. $10^4$-$10^7$ W.cm$^{-2}$ for 0.0001-0.1 ms). No additional al-
loying elements are incorporated. Since the bulk of the material is unaffected by the laser, a
large heat sink is provided for the subsequent rapid cooling of the melted surface and cooling
rates in the region of $10^4$-$10^8$ K.s$^{-1}$ can be obtained. This can result in non equilibrium
microstructures which may confer substantial increases in hardness and wear resistance, the
application areas which have received the most attention, particularly for steels, cast irons
and aluminium alloys. Figure 1 [1] shows the power density/interaction time regime for LSM
(glazing) compared with other laser processes of interest.

A schematic diagram of the experimental arrangement for LSM treatment is shown in
figure 2 [1].

Enhanced corrosion properties can be produced by LSM as a result of altering the sur-
face composition, changing the microstructure or by redistribution of impurities and second
phases [3-18]. In general the non equilibrium surface microstructures produced result in
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Fig. 1. — Regimes of laser irradiation intensity and interaction time for various types of materials processing [1].

Fig. 2. — Experimental arrangement for laser surface treatment [1].

finer, more uniform structures with superior homogeneity compared with conventional surfaces.

For example, redissolution of MnS inclusions following LSM resulted in increase in the critical pitting potential of type 304 stainless steel in 0.1 M NaCl solution [3, 4, 7-10]. (Deterioration in resistance to pitting in Mo-bearing ferritic stainless steels at low Mo contents as a result of LSM has been observed [8]).

LSM has been applied to sensitised type 304 [11, 13, 16] and type 316 [18] stainless steels
where redissolution of Cr during LSM offers a successful means of overcoming intergranular corrosion associated with weld decay. Hence there are good prospects for an in situ surface engineering technique for the upgrading of welded components at risk from this form of corrosion.

Cr enrichment of the passive layer as a result of LSM has been observed on Fe-19% Cr alloy [5] and in Type 304 stainless steel [12] subject to excimer laser radiation where enhanced resistance to pitting was observed.

1.3 LASER SURFACE ALLOYING (LSA). — A definitive bibliography (to 1983) and review (to 1985) of LSA has been published by Draper and Ewing [19] and by Draper and Poate [20], respectively. Coating strategy is achieved by incorporating additional alloying elements in the surface melt region produced by the laser. The laser power density and interaction time regime (Fig. 1) is thus similar to that required for LSM and the experimental arrangement (with allowance for alloying element incorporation) is also similar. The objective of LSA is to alloy completely added elements into the near surface while avoiding alloying element loss by vapourisation or dissolution in the substrate. Typical melt depths are 150 - 2000 μm.

Enhanced surface properties such as wear and corrosion resistance [1, 3, 6, 7-9, 15, 19-32] can be achieved while bulk substrate properties remain unaffected. From a corrosion resistance point of view, compositional uniformity in the surface alloyed layer is desirable and this is normally achieved within each melt track as a result of turbulent mixing effects [20]. Compositional inhomogeneity may remain at the overlaps between adjacent tracks on the surface. Alloying elements can be made available to the surface during LSA by a large number of means which include ion implantation, electrodeposition, vapour deposition, flame spraying, sputtering, preplaced foil or powder techniques (in which a coating or layer is present prior to processing) and techniques such as the blown powder process where metal powders are elutriated into the laser melt zone by inert gases and which allow for continuous processing.

McCafferty, Moore and co-workers [3, 7] laser surface alloyed Cr into low carbon steel (AISI 1018) and found a uniform Cr distribution in the alloy layer. Potentiodynamic polarisation tests in deaerated 0.1 M Na₂SO₄ solution showed that increase in Cr content in the alloyed layer resulted in a decrease in the critical current density required for passivation \( i_{\text{crit}} \) and a decrease in the current density in the passive range \( i_{\text{pass}} \). LSA of Cr and Ni in AISI 4140 plain carbon steel [8] to give 29%Cr and 13%Ni in the surface layer resulted in a reduction in \( i_{\text{crit}} \) and \( i_{\text{pass}} \) compared with type 304 stainless steel on potentiodynamic polarisation in deaerated 1 N H₂SO₄ solution. Mazumder and Singh [23] found enhanced corrosion resistance compared with type 304 stainless steel for LSA of Cr and Ni in plain carbon steel on potentiodynamic polarisation in 3.5 wt% NaCl solution. McCafferty and Moore [9, 22] alloyed Mo into type 304 stainless steel and found increase in critical pitting potential with Mo content on potentiodynamic polarisation in 0.1 NaCl solution. The critical pitting potential for surface alloys containing 3% Mo was comparable with that of bulk 316 alloy while surface alloys containing 9% Mo showed no pitting breakdown up to the oxygen evolution potential.

Corroborative results have been achieved for LSA of Cr [15, 27, 28], Ni-P [29] or Cr and Ni [15, 24, 28, 31, 32] in plain carbon steel substrates. Weston and Wright [28] point to the effect of cracks in the alloy layer in lowering corrosion resistance. Meletis and Hochman [25] report LSA of Nb in types 316 stainless steel.

1.4 LASER SURFACE CLADDING. — The objective in laser cladding is to produce a relatively thick and homogeneous overlay of coating material on the substrate with a sound interfacial bond and a minimum of contamination of the coating material with elements from the
substrate [1, 33]. Hence the process is carried out in a regime (Fig. 1) where the power density is lower and the interaction time is longer than in LSM or LSA (eg. $10^3 - 10^5$ W/cm$^2$ for 0.1 to 1 s). The coating material may be supplied to the laser melt zone in a variety of ways which include preplaced powder and blown powder techniques as well as laser chemical vapour deposition (LCVD), laser physical vapour deposition (LPVD) and laser enhanced electrodeposition routes.

The arrangement for laser cladding by the blown powder technique is shown in figure 3. Essentially, cladding material in the form of powder is propelled by inert gas into a region where a laser generated molten pool of powder material can be formed which is subsequently deposited on a traversing substrate surface. A reflective dome can be used to improve process efficiency and to provide a means of inert gas shrouding to prevent oxidation.

![Diagram](image)

Fig. 3. — Schematic arrangement for laser cladding by the blown powder technique [1].

Variation on this technique (to be discussed in Sect. 3) includes the use of multiple hoppers [24] for the in situ mixing of coating alloy of any given composition from powders of the elements making up the coating alloy.

Laser cladding on steel with reported enhanced corrosion resistance in a variety of applications includes Cr-Ni alloys on 1016 plain carbon steel [23], Stellite alloys on steel oil well valves [34], 50Ni/50Cr alloy on AISI 347 stainless steel [35] and NiCrSiB alloys on steel [36].

A number of workers have employed laser remelting of clad layers on steel produced by other coating techniques for the subsequent removal of porosity and have reported enhanced corrosion performance. These include subsequent laser treatment of plasma sprayed NiCr [37], thermally sprayed stellite alloy [38] and Ni alloy [39] coatings, electroless Ni-P coatings (Nd-YAG laser) [40] and Mo plasma sprayed coatings on stainless steels [41].

Laser remelting of ceramic layers for high temperature corrosion and oxidation resistance has been investigated. Laser remelting of silica layers predeposited by SOL-GEL or PAVD technique resulted in coatings with long term protection of 20/25 Nb steel substrates exposed
to CO₂ based gas mixtures at 700 °C or 825 °C and to Incoloy 800H substrates exposed to sulphidising coal gasification (CGA) atmospheres at 450 °C [42-46]. Fused oxide coatings of silica and alumina deposited by means of the blown powder process have also been investigated [47, 48] with good resistance to CGA environments at 450 °C following removal of porosity with a subsequent LSM treatment. This has included work on the effect of deposition on inclined substrates. Surface remelting of plasma sprayed Y₂O₃ stabilized ZrO₂, including the application of excimer laser processing, has also been investigated [49].

2. Investigation of variable composition laser cladding as a diagnostic technique in alloy development.

The principle of the production of laser melt tracks of variable composition for alloy development purposes via the control and incorporation in the laser melt zone of metered mixtures of metal powders has been demonstrated in our laboratories for the model system Co-Ni-Fe.

Co, Ni and Fe powders of approximate size 150 microns were fed into the fusion zone of a CO₂ laser from separate hoppers under microprocessor control so that the relative proportions of the elements were controlled. Oxidation was prevented by Ar gas shrouding in conjunction with a reflective dome which minimized energy losses. Laser process conditions were controlled so that liquid phase alloying between the constituents took place under the action of the laser before impingement and solidification of the material as a melt track of width approximately 3-5 mm on a steel substrate. A schematic diagram of the experimental arrangement is shown in figure 4.

![Experimental arrangement for the production of laser melt tracks of variable composition.](image)

By controlling the proportions of the constituents, the whole range of composition in the ternary system was produced on a single steel substrate in a single, speedy operation (Fig. 5).
For example, track 1 (containing zero Ni throughout) commenced as pure Fe and the percentage of Co was increased linearly with distance until the track finished as pure Co. Track 2 contained 10 wt% Ni throughout with the proportion of Fe decreasing linearly with distance and with the balance made up by addition of Co. Track 3 contained 20% Ni with a similar variation of Fe and Co with distance along the track. Subsequent tracks contained 40%, 50%, 60% Ni, etc with corresponding variation in Fe and Co until the whole composition range had been studied. The resulting single sample thus contained in a form ideal for further investigation the complete range of possible compositions for the alloy system.

The laser employed was a 2 kW Electrox CO₂ CW laser operating at a typical power level of 1.5 kW with a specimen movement rate of 4 mm/s and spot size of 4 mm, giving a power density of $6 \times 10^3$ W/cm² and an interaction time of 1 s.

Variation on the technique includes an increased resolution of composition (for example, once a candidate composition range has been identified, variation of Co and Ni could be accomplished in tracks in which the variation of Fe was 1%) and the ability to investigate low level alloying by the use of prealloyed powders. Tracks can be heat treated to develop equilibrium microstructures or tested in the as deposited condition. An important aspect of this work is that conditions for the deposition of thick surface layers via the laser surface cladding technique are simultaneously determined. Evaluation techniques that are applicable to variable composition laser melt tracks include the determination of hardness, wear resistance, microstructure and corrosion properties.

3. Conclusions.

1) Despite outstanding advantages of laser surface treatment techniques for enhancement of corrosion resistance, these techniques are not yet widely employed because of the small area that can be processed in a single pass. Development of large area treatment techniques would dramatically change this situation.
2) A brief review of laser surface melting, laser surface alloying and laser cladding techniques on steel substrates has shown that the potential for the production of corrosion resistant coatings by these means is considerable.

3) A new alloy prototyping technique based on laser cladding via the blown powder technique which has been developed at Liverpool has been outlined. This should provide a valuable tool in work aimed at the development of alloys of enhanced corrosion resistance and other significant materials properties. An advantage of the technique is that deposition parameters via laser surface cladding for any composition within the alloy system in question are simultaneously determined.

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