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Generation of hardened steel surfaces with adjustable roughness by means of a pulsed electron beam

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ABSTRACT

In this work we are utilizing a Pulsed Cold Cathode Electron Beam (PCCEB) for steel surface treatment that presents distinct properties as compared to other beam sources for rapid heating of metal surfaces. The possibility of producing different surface transformations like, melting and solid–solid transformations by adjusting the gun control parameters is demonstrated. It is shown that for the same base material, different pulse conditions give rise to distinct structures, a roughened or smooth martensitic surface or a smooth pearlite layer several microns thick. For the liquid–solid transformation, it is shown for the first time that with an adequate impurity content the instability onset threshold (giving rise to a rough topography) is different from the melting threshold. Hence, by controlling the discharge parameters, it is possible to obtain different topographies with similar crystallographic structure and hardness, allowing an adjustment of the final roughness to the particular application requirements.

1. Introduction

In the last few decades, new methods have been developed for surface modification of metallic materials which are based on the use of intense pulsed laser [1,2], electron and ion beams [3–5]. Irradiation induces superfast heating in the surface layers giving rise to melting and evaporation, followed by superfast cooling of the surface layer and the consequent development of metastable phases. The common feature of all of these processes is the production of certain thermal cycles in small, highly localized regions on the surface of the sample, which then takes on new properties that allow it to cope better with wear, fatigue, corrosion, and surface hardening, while maintaining most of its other original properties. Many beam irradiation techniques for steel treatment consist on a rapid heating of the surface into the austenitic region [6]. Due to the high rates of heat transfer, steep temperature gradients are set up which result in rapid cooling by conduction. This causes the transformation from austenite to martensite without the need for external quenching. This self-quenching occurs as the cold interior of the sample constitutes a sufficiently large heat sink to quench the hot surface by heat conduction to the interior at a rate high enough to prevent pearlite or bainite formation at the surface, resulting in martensite formation.

The effects of Pulsed Electron Beam Surface Treatment (PEBST) are similar to surface treatment using pulsed lasers, but electrons provide unique capabilities that allow it to avoid many problems intrinsic to pulsed laser technology, including poor energy coupling to metals, inefficient in-depth treatment, edge effects, and high cost.

Surface treatments with pulsed electron beams use sources with electron energies typically in the tens of keV, pulse durations ranging from submicroseconds to tens of microseconds and fluencies between 1 J/cm^2 and tens of J/cm^2 [3,5,7–13]. Two clearly distinguished regimes have been recognized [14,15], a subcritical one in which the surface melts but does not vaporize and a supercritical regime in which the fast evaporation gives rise to the appearance of a Richtmyer–Meshkov (RM) instability and a subsequent crater formation is detected [15]. In the subcritical regime the RM instability is suppressed by the surface tension and surface smoothing is achieved [14].

In previous works it was shown that different instabilities may develop originated in a Bénard–Marangoni (BM) convection, that give rise to surface roughening in the subcritical regime [3,16]. It was shown that depending on the impurity content a hydrodynamic instability may develop when the surface tension of the melt decreases with the temperature. If this happens, the resulting structure freezes in the solid state giving rise to a roughened surface at the micrometer scale. The mechanisms involved are different from those appearing with other fast treatment techniques based on lasers or electron beams where the onset of instabilities is related to rapid evaporation [15,17]. The BM instabilities depend on the material composition and impurities, they do not appear in pure iron and have been reported in AISI4140 with large sulfur content [3,16]. In all prior cases the instability developed very fast (few microseconds) and was always observed if the surface was melted. The amplitudes of the undulations in the liquid were so large that the melted pool

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dewetted the solid substrate, resulting in a very peculiar structure (spinodal dewetting) with unquenched material exposed [3].

This work is centered in heat surface treatment of steels by means of a Pulsed Cold Cathode Electron Beam (PCCEB) that delivers tens of joules in tens of microseconds [3,5]. The electron gun produces a low pressure glow discharge that impinges the sample producing particular topographies and structures. Depending on the control parameters like gas pressure, source voltage or cathode–sample distance it is possible to induce surface phase transformation in the solid and liquid state.

In this paper it will be shown that by an adequate choice of the composition (impurity content) the onset of the instability can be controlled by the discharge parameters, allowing a superfast cooling of the surface layer with a smooth or rough topography at will. The fact that the instability is not as abrupt as in prior works permits the generation of roughened surfaces without the dewetting onset, hence exposing to the ambient only quenched (hardened) phases. Hence a new threshold in the treatment fluency appears in the subcritical regime that separates the onset of the melt surface instability from the surface smoothing response.

AISI4140 steel irradiated by PCCEB is investigated in order to explore the influence on surface properties for different irradiation conditions. Modifications in the treated material include macroscopic changes in the scale of micrometers (rough or smooth), microscopic changes, modification of the crystallographic structure, different temperature field and consequently hardness profile changes. AISI4140 steel was chosen because of its relatively simple composition and structure, its widespread industrial use, and the relevance of comparing with prior results [3] in order to show the possibility of controlling the resultant surface roughness with the treatment conditions for adequate impurity content.

As the surface is cooled very fast below the martensite start temperature (Ms), a diffusionless phase transformation develops achieving an extremely hard brittle martensite surface layer which is desirable where a hard, wear resistant case is required, leaving the bulk properties of the material such as toughness and ductility unchanged. The novel structures produced and the advantages of obtaining the same microstructure with different topographies are discussed.

In Section 2 the experimental and numerical techniques are described, as well as the treatment conditions. In Section 3 the results are presented on microstructure, crystallographic and hardness characterization (on most representative samples) and temperature field simulation, including a discussion of the results obtained. A correlation between the hardness and the microstructure is described for each region of the heat affected zone and for different PEBST conditions. In Section 4 (conclusion) a comparison is made, between the two types of topographies obtained: smooth and roughened.

2. Experimental and numerical techniques

Pulsed Cold Cathode Electron Beam (PCCEB) used in this work is described in detail in Ref. [18]. It provides intrinsic in-depth energy deposition and an inert helium atmosphere, low capital cost of hardware, and high (over 35%) wall-plug-to-surface electrical efficiency. This is also an advantage of PCCEB with respect to electron beams produced by heated filament sources. The energy coupling of electron beams to a material is independent of the surface preparation and very insensitive to surface temperature or crystallographic phase. The beam power density is controlled experimentally by several parameters. One is the total energy delivered by the gun, which is set by the charging capacitor (fixed at 90 nF) and the charging voltage (which can be varied between 13 and 30 kV). Another control parameter is the He gas pressure, which determines the peak current and the pulse duration for a given voltage. The third one is the sample distance from the cathode, which determines the electron-beam diameter at the sample position, because the beam starts with the cathode diameter and converges to a minimum at a focal point (that depends on the voltage and current), due to the cathode concavity (R=200 mm) and the focusing action of the Lorentz force. For different discharge conditions, the power density was measured by means of the knife-edge method [5] (consisting of measuring the beam current intercepted by a sharp edge at different positions), and it was found to fit to a Gaussian current density spatial profile plus a small constant background current density [3].

The samples used were hypoeutectoid commercial grade steel, AISI 4140 type (ferrite pearlite). The concentrations are listed in Table 1. The samples were surface treated by means of a Pulsed Cold Cathode Electron Beam (PCCEB). A detailed description of the gun can be found elsewhere [3,5]. It produces a high voltage high peak current pulse that is focused in a controlled manner on the sample surface.

The beam energy is deposited in a very thin surface layer and the heat penetrates by diffusion, melting several micrometers of material. After rapid cooling towards the substrate, a modified spot appears with a characteristic spatial pattern.

Sample A is the base material that was normalized according to the conventional cooling cycle for AISI4140 steel alloy given by Ref. [19], standardizing in this manner the same departure structure for all the surface treated samples.

For most of the characterizations a single PCCEB shot is applied to samples B to E with different cathode voltage drop according to the details given in Table 2. The cathode–sample distance was held at 170 mm and with the same He partial pressure, 40 Pa. V_{source} is the power supply set voltage, V is the cathode voltage (that differs from the source voltage due to the pulse shaping circuit), I_{max} is the pulse peak current obtained at this pressure; $P_{max}=(V\,I)_{max}$. τ_P is the power pulse width measured at the FWHM. The energy is calculated by integrating the measured instantaneous power; the fluency is obtained from the integration of the product of the cathode voltage and the current density at the beam center. Different results are obtained not only for the different discharge conditions but also within a given spot along the radius as will be discussed later. The single shot studies allow the evaluation of the different structures appearing within the treated area as a function of the distance from the beam center. For the X-ray studies and in other cases specified in the text, multiple partially overlapping pulses were applied in order to cover a larger treated area. In such cases the overlap was performed in such a way that only the central structure is retained (see below).

The surface characterization was performed with optical and scanning electron microscopy (SEM), atomic force microscope (AFM), and X-ray grazing incidence (to correlate the effect of treatment parameters with microstructure). Nanoindentations are performed on different structures obtained showing a strongly hardened material for the transformation reaching the melt.

For cross section metallographic observations the specimens were embedded in an acrylic resin that stiffens at room temperature. Previously, the samples were nickel electroplated to produce a protective coating for the polishing. After cutting, the classical grinding and polishing stages are performed at least down to the use of 0.05 μm Al_2O_3 slurry. After every sequence of polishing, the specimen is rinsed several times with alcohol, and then dried with a flush of dry nitrogen. Finally, the cross sections were etched with nital (2 vol.% nitric acid in ethylic alcohol), during 3 s, in order to reveal the structure. For the front view observations a one second immersion in nital 2% was performed, when necessary, to reveal the structure.

The resulting structure was observed by optical and scanning electron microscopy (SEM), both in a top view and a cross section. The sample micro-topography was analyzed by means of an atomic force microscope (AFM).

Table 1
Chemical composition of steel used in this study

Element	C	Mn	P	S	Si	Cr	Mo
wt.%	0.36	0.79	0.012	0.006	0.18	1	0.19