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Grain refinement, hardening and metastable phase formation by high current pulsed electron beam (HCPEB) treatment under heating and melting modes

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ABSTRACT

High current pulsed electron beam is a recently developed technique for surface modification. The pulsed electron irradiation introduces concentrated energy depositions in the thin surface layer of the treated materials, giving rise to an extremely fast heating and subsequent rapid cooling of the surface together with the formation of dynamic stress waves. Improved surface properties (hardness, corrosion resistance) can be obtained under the "melting" mode when the top surface is melted and rapidly solidified (10^7 K/s). In steels, this is essentially the result of nanostructures formed from the highly undercooled melt, melt surface purification, strain hardening induced by the thermal stress waves as well as metastable phase selections in the rapidly solidified melted layers. The use of the "heating" mode is less conventional, combining effects of the heavy deformation and recrystallization/recovery mechanisms. A detailed analysis of a FeAl alloy demonstrates grain size refinement, hardening, solid-state enhanced diffusion and texture modification without modification of the surface geometry.

1. Introduction and state of the art

The interaction of intense-pulsed energetic beams such as ion, electron and laser beams with material has been studied extensively recently [1–4]. These pulsed beam techniques allow high energy deposition at a short time within narrow depths near the material surfaces. Among them, the low energy high current pulsed electron beam technique (LEHCPEB) is fairly recent [2,4]. As a high-power charged particle beam, LEHCPEB exhibits essential advantages over pulsed laser and ion beams by its efficiency, simplicity and reliability. The pulsed electron irradiation induces (i) a rapid heating and cooling of the surface together with (ii) the formation of thermal stress waves [4]. As a result, improved surface properties, often unattainable with conventional surface treatment techniques, can be obtained fairly easily. This is particularly true for tribological [2,4] and corrosion properties [5–7]. The material surfaces were treated using a Nadezhda-2 type LEHCPEB apparatus [4,8]. Some procedures for characterization by EBSD, XRD and TEM were detailed in Refs. [7–12].

Fig. 1 illustrates the effect of the LEHCPEB treatment for improving the corrosion resistance (Fig. 1a) and wear resistance (Fig. 1b) in the case of steels [6]. The surface properties revealed in Fig. 1 must be determined by the final structure-phase states generated by the LEHCPEB thermo-mechanical treatment. However, limited information is available on the exact metallurgical phenomena controlling the microstructure modification under LEHCPEB. The aim of the present paper is to review some recent findings obtained when characterizing the surface of LEHCPEB treated materials. In particular, are detailed here some of the features observed when treating with or without melting of the top surface (i.e. *melting and heating modes*). The distinction between these two modes is important to get the full potential of this technique. Therefore, the examples of surface modifications given here are within two separate sections related to these two treatment modes.

2. Examples of surface modifications

It is generally accepted that three different zones are usually observed in the surface depth of LEHCPEB treated samples [4,8–10]. Present at the top surface is a zone that has been melted and subsequently solidified rapidly. It is often a few μm in thickness but can be avoided, in particular for high conductivity alloys, when the energy provided by the electron beam is not sufficiently high [13]. Below is found a heat-affected zone (HAZ) that extends generally

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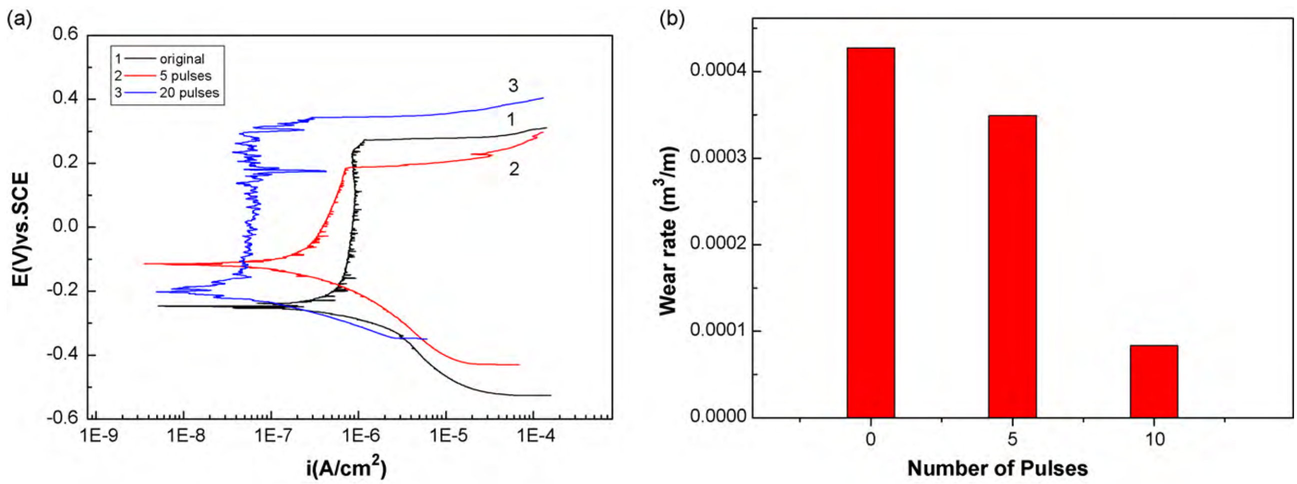

Fig. 1. Potentiodynamic polarization curves (a) and pin-on-disk wear rates (b), showing the effect of the number of LEHCPEB pulses for improving the corrosion resistance of an AISI 316L stainless steel [6] (a) and wear resistance of an eutectoid carbon steel [unpublished results] (b).

over a few tens of μm. Finally, a last zone resulting from the propagation of the stress wave may be present at depth far exceeding the heat-affected zone. After sufficient pulses of LEHCPEB (typically 15–20), the material hardness can be strongly increased in this zone over hundreds of μm [9].

2.1. Modifications under the melting mode

Several studies have reported on the formation of craters at metal surfaces induced by pulsed ion beams [14,15]. The formation mechanism appears simple: it is the non-homogeneity of the ion distribution within the ion beam that leads to the crater formation. However, things are different in the case of a pulsed electron beam where the momentum of a single electron is very low and the beam cross-section is homogeneous. A detailed analysis of the crater formation in various LEHCPEB treated steels has recently been published [16]. The onset of melting starts locally just below surface and, due essentially to the expansion of volume associated to the passage from the solid towards the liquid as well as possible vaporization of the melted mater, explosion occurs when the thickness of the solid layer confining this pool of melt cannot sustain anymore the expansion forces. It was observed that heterogeneities such as carbides [6,7,16,17] or intermetallic precipitates [5–7] often serve as nucleation sites for the eruption events. It was also clearly demonstrated that the eruption events tend to expulse the precipitate matter [5] and break them up into smaller parts [17]. This break-up of the precipitates and their subsequent dissolution reduce the level of segregation in the re-solidified layer [17]. Therefore, with increasing the number of pulses and melt thicknesses, less nucleation sites are available and the crater formation mechanism becomes less effective [16]. This leads to the formation of a homogeneous – almost crater free – surface after a sufficient number of pulses; as was observed for the 316L [18], D2 [17] and carbon steels [16] as well as a NiTi shape memory alloy [19]. The so-formed homogeneous layers were demonstrated to improve the corrosion properties of these dual phase alloys [5–7]; as illustrated in Fig. 1 for the 316L steel [6].

Another interesting aspect concerning the melted layer is the change in phases resulting from the rapid solidification in steels. Austenite usually should transform after solidication into martensite because of the very high cooling rate (∼10^7 K/s). However, only the austenite phase was retained in the top surface melted layer after 20 pulses in the D2 steel [17]. The dissolution of the carbides [17] together with the ultra fine grain size of the matrix grains [17,20,21] were considered to be important factors for the stabilization of the austenite phase. Under the “melting” mode, the LEHCPEB treatment often induces the formation of an ultra fine grained layer formed by rapid solidification, such as in the D2 steel and NiTi alloys [17,19]. These ultra fine grains are one of the reasons for surface hardening. In addition, the formation of martensite in steels [17,18] and the presence of structural defects induced by the formation of thermal stresses also play an important role for increasing hardness and improving wear resistance [18]. The strain hardening associated with the thermal stress is witnessed by the observation of slip traces (Fig. 2) or in some cases, depending on the stacking fault energy of the material, by the observation of deformation twins (316L steel) [22].

2.2. Modifications under the heating mode

Compared to the previously investigated alloys, the two types of Fe–40Al (at.%) intermetallic alloys investigated in this section were treated below the onset of melting. Further information concerning these alloys can be found elsewhere [23,24]. The initial extruded bars were obtained by extrusion from milled (with Y_2O_3 addition at the milling stage) [25–28] or atomized [25,26] FeAl powders. The extrusion temperatures (1250 °C and 1100 °C) and the result-

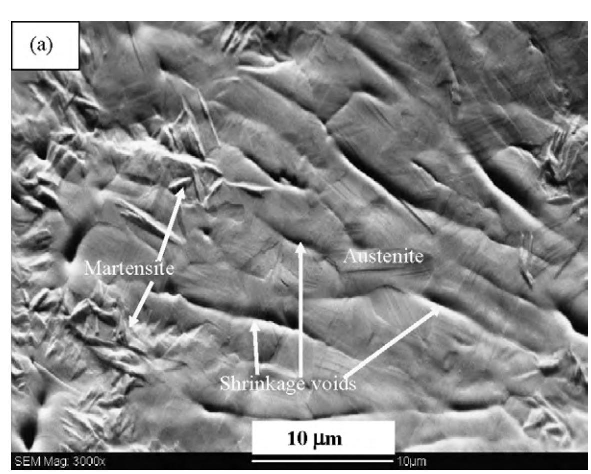

Fig. 2. SEM image illustrating features revealed at the melted top surface of a D2 steel treated for 5 pulses: the presence of martensitic needles + residual austenite containing intense slip traces.

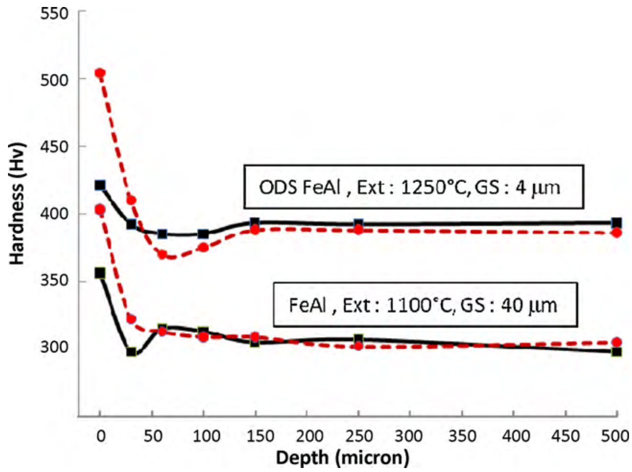

Fig. 3. Evolution of the cross-section hardness of two FeAl alloys after HCPEB treated for 2 pulses: (i) with (rounded symbols) and (ii) without (squared symbols) the presence of previously deposited graphite layer (solid state surface alloying).

ing average grain sizes (4 μm and 40 μm) obtained for these ODS and conventional FeAl alloys are recalled in Fig. 3. The samples were treated for 2 pulses with or without the presence of pre-deposited surface graphite layer. Fig. 3 shows the evolution of the micro-hardness as a function of the depth from the top surface. Because of a smaller grain size and the presence of the oxide dispersion, the bar of the ODS alloy is characterized by a higher hardness (about 390 HV) than the conventional one (about 300 HV) [26,27]. In both cases, even after only 2 pulses, an increase of about 50 HV in hardness is associated with the LEHCPEB treatment at the surface of the FeAl sample (curves with squared dots). It is well established that the LEHCPEB treatment leads to the formation of quenched-in vacancies [29]. The presence of vacancies, which are known to affect the strength of FeAl [30–32], is likely to induce some increase in hardness. In addition, some hardening observed in this intermetallic is also due to the combination of grain refinement and texture modification [13]. Indeed, as illustrated in Fig. 4, EBSD analysis on a 20 pulsed FeAl sample has revealed that the repeated deformation and dynamic recovery/recrystallization processes occurring during the LEHCPEB treatment induce the formation new grain boudaries together with a significant modification of the surface texture [13]. While the initial extruded bar was characterized by a ⟨1 1 0⟩ fiber texture [23] (Fig. 4b), new sub-grains are created (Fig. 4c) and the grains have blueish colors coresponding to near ⟨1 1 1⟩ orientations (Fig. 4d) after the HCPEB treatment [13].

Fig. 3 also demonstrates solid state surface hardening and rapid surface alloying effects associated with the LEHCPEB treatment when a graphite layer was previously deposited on the surface. Indeed, a strong effect – an increase of more than 100 HV – is

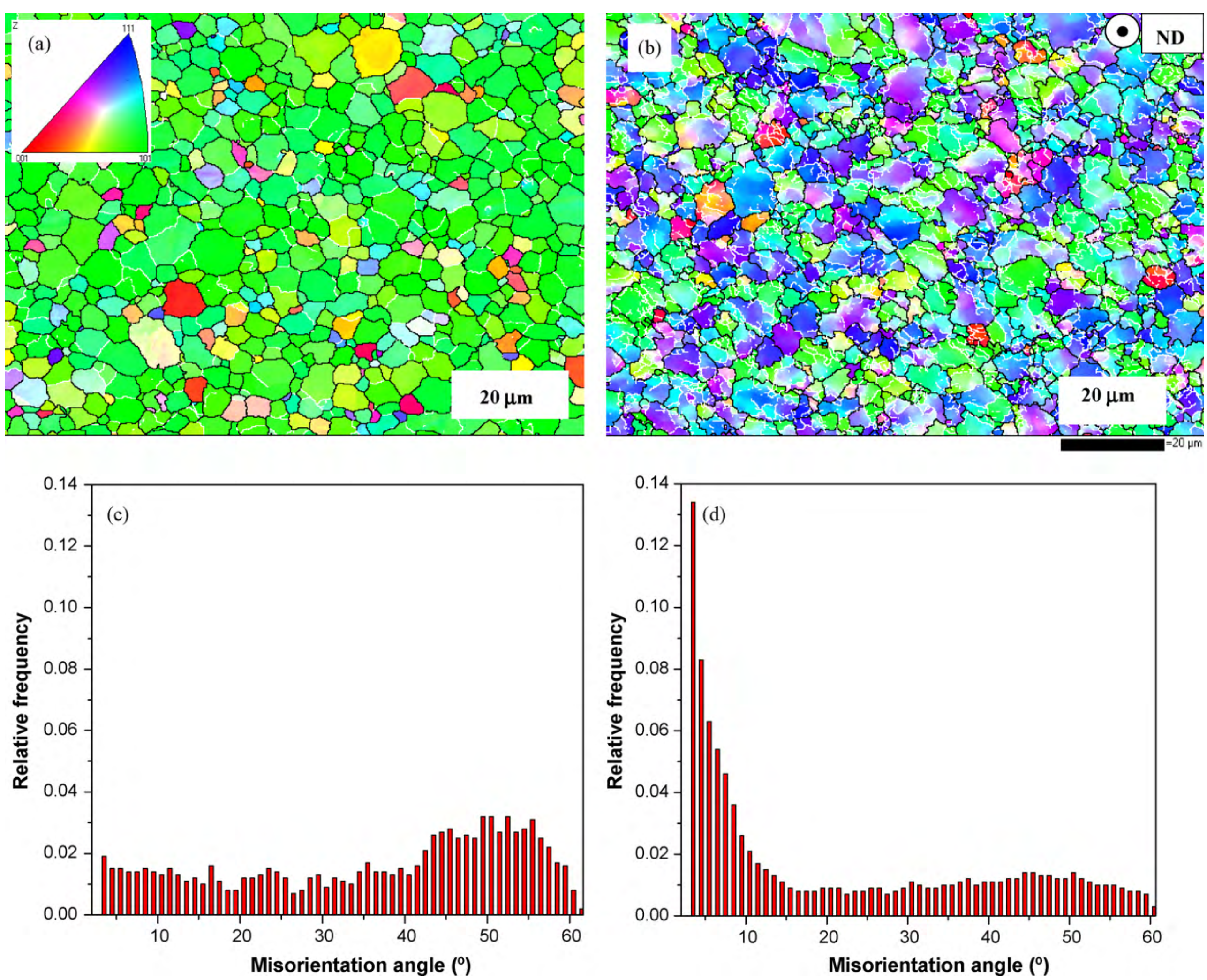

Fig. 4. EBSD analysis showing the evolution of microstructure and texture (a and b) as well as grain boundary misorientation (c and d) of a FeAl alloy in the as-received (a and c) and 20 pulsed HCPEB treated (b and d) states.

associated with the thermally enhanced solid-state diffusion of C on hardening the surface (curves with rounded dots). This further increase in hardness can be due to the combination of the intrinsic effect of C in the FeAl cell – as it is established that C atoms do strengthen the atomic binding [32] – and the precipitation of carbides [33].

3. Summary and conclusion

In summary, it is clear that the LEHCPEB irradiation induces drastic temperature gradients in the surface of the material and that, concomitantly to the thermal effect, the pulse electron beam creates a dynamic stress field that causes intense deformation at the material surface and sub-surface. Therefore, the high potential of the LEHCPEB technique can be better achieved by a good control of the processing parameters in order to treat the sample surface under the so-called *melting* and *heating* modes. The number of pulses of LEHCPEB treatment is also shown to affect the microstructure in different manners.

Under the *melting mode*, after an initial stage of crater eruption, the formation of a homogeneous layer is obtained when increasing the number of pulses. This layer can improve drastically the corrosion properties. In the case of steels, the formation of metastable phases can be triggered. For example, the retention of the metastable austenite was observed at the top surface of a D2 sample treated for high number of pulses.

Comparatively, the *heating mode* has been shown to provoke (i) hardening and (ii) carbon stress enhanced rapid solid state alloying when the material was previously covered by a graphite layer. The heavy deformation induced by the repeated pulses also induces the formation of new grain boudaries together with a significant modification of the surface texture state without any modification of the overall sample surface shape.

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