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Formation of large grains by epitaxial and abnormal growth at the surface of pulsed electron beam treated metallic samples

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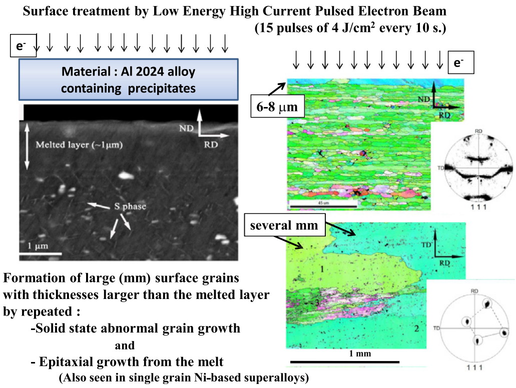
HIGHLIGHTS

- Extremely large (mm) grains were obtained at the surface of pulsed electron beam treated metallic samples.
- These millimetric grains formed in Al alloys by the combination of epitaxial growth and abnormal grain growth.
- Epitaxial growth leads to solidified grains having sizes and crystallographic orientations inherited from the substrate.
- The driving force for abnormal grain growth is the difference in defects generated at each irradiation on cooling.

GRAPHICAL ABSTRACT



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ABSTRACT

The aim of the present work was to clarify that the rapid solidification process involved during surface pulsed beam treatment does not necessarily leads to the formation of nano or ultrafine grains. To demonstrate this, two types of metallic materials (a slightly deformed 2024 aluminum alloy and nickel based single grain superalloys) have been irradiated 15 times by high current pulsed electron beam. Epitaxial growth was triggered during the re-solidification of the superalloys, leading to the formation of a deformed single grain. Thanks to the repeated combinations of epitaxial growth and solid state secondary recrystallization, the pulsed electron beam treatment has led to the formation of large (millimetric) grains, that were also 4 to 5 times thicker than the depth of the melted zone, at the surface of the aluminum alloy.

1. Introduction

Initially developed in Tomsk (Russia) at the Institute of High Current Electronics [1–3], the high-current electron beam (HCEB) technology has now attracted much attention for material surface modifications.

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The pulsed electron beam technology employed in the present manuscript uses low energy (up to about 40 keV), high-current (up to about 50 kA) electron beams of microsecond durations that can treat a rather large surface area of about 100 mm in diameter. For surface modifications, this type of extremely short pulse treatment is often repeated over 5 to 50 cycles and generally induces the formation of three successive zones in the irradiated samples. If the energy imparted to the sample is sufficiently high, the very top surface (few microns) consists generally of a melted and rapidly solidified layer from which some light element may have evaporated [4]. Below is present a heat affected zone (~10 μm) where solid-state phase transformations and recrystallization may occur [5,6]. Finally, much deeper within the surface (~100 μm), can be present a zone that is affected by the generated stresses and which can be hardened by plastic deformation and the presence of structural defects such as vacancies and dislocations [7–10] as well as by the formation of stress induced martensite [11,12] or deformation twins [13,14].

After its development in Russia, this technology - and very close ones - has spread towards Asia (China, Japan, Korea), Europe and America. Because of its pulsed nature and the relative low energy of the electron beam that spreads in one shot over a large area, this technology has received a number of different types of acronyms depending of the types of rigs and applications: low energy high-current electron beam (LEHCEB) [1–3,15], (low energy) high current pulsed electron beam ((LE)HCPEB) [5,8–12,16], pulsed electron beam (pulsed EB) [17] as well as large-area electron beam (LAEB) [18–20] or large pulsed electron beam (LPEB) [21,22].

When the top surface is melted, because of the heat extraction towards the sub-surface, the solidification process can lead to solidification rates as high as 10^7 K/s [15,23]. Under such rapid solidification conditions, metastable phase transformations can occur which may harden the top surface [24,25], Also, when sufficient number of pulses are used in multiphase alloys (in particular those reinforced by precipitates), the repeated melting can homogenize the chemistry of the top surface, which generally leads to improved corrosion properties [26–30]. It has also been claimed in several contributions that the rapid solidification process inevitably leads to significant grain refinement down to the “nanometer” or, more rigorously, the sub-micrometer range. Indeed, ultrafine and nanoscale precipitates and domains [31–33], ultrafine and nano-grains [34–36] and nanostructures [37–39] are regularly reported on a wide variety of metals. Comparatively, Samih et al. [40] reported the formation of large grains, up to 50 μm in diameter, at the surface of a AISI 420 martensitic stainless steel. This clearly means that the formation of ultrafine grains is not necessarily associated with such type of treatment. To demonstrate this, from our understanding of the HCPEB surface modifications, we present here an analysis of the surface modification at the surface of an aluminum alloy for which the formation of large grains, having a size reaching the mm range, has been triggered. At the light of some analyses carried out on HCPEB treated single crystal superalloys, the formation of these very large grains will be discussed in terms of epitaxial growth of the melted liquid from the substrate followed by solid state abnormal grain growth.

2. Experimental procedure

2.1. Materials selection

2.1.1. The 2024 alloy sheets

A commercial 2024 Al alloy was used in this investigation. This alloy is an Al-Cu-Mg based alloy used essentially for aircraft application in the form of plates or sheets. The exact chemical composition of the 2024 alloy used in the present investigation is as follows (wt%): Al-4.3%Cu-1.5%Mg-0.6%Mn-0.3%Fe-0.3%Si. This 2024 alloy was delivered to us in the form of 10 mm thick rolled sheet. Then, the sheet was treated under the so called T9 condition - homogenized, aged then cold worked - before the HCPEB surface treatment. The alloy was homogenized at 500 °C for 1 h followed by water-quenching. Subsequently, the sheet was aged at 200 °C for 3 h and cold rolled to a reduction ratio of 15% by four successive passes carried out along the same rolling direction (RD).

2.1.2. The Ni-based single grain superalloys

The Ni-base superalloys used here are the so-called CMSX2 [41] and AM1 [42] alloys. The nominal chemical compositions of the AM1 and CMSX2 alloys are (wt%): Ni-7.5%Cr-5.5%W-8%Ta-5.3%Al-6.5%Co-1.2%Ti-2%Mo and Ni-8%Cr-8%W-6%Ta-5.6%Al-4.6%Co-1%Ti-0.6%M [43]. These alloys have been developed by the aircraft industry for directional solidification as single grain turbine blades. Thus, due to the absence of grain boundary, they could contain a high fraction of ordered γ′ precipitates dispersed in a face centered cubic (fcc) Ni based γ matrix without any other carbide or additional grain boundary strengthening phase. The superalloys were grown as single grain using the Brigman method in the shape of cylindrical rods having a diameter of 10 mm. The growth direction (GD) was along their [001] crystallographic direction. After solidification, they received a conventional optimization treatment in order to optimize the volume fraction and distribution of the coherent γ′ precipitates within the γ-Ni-based matrix [42]. Because of their high volume fraction (65 to 70%) and their coherency with the γ matrix, these γ′ precipitates adopt a cuboidal shape (with a mean edge size of about 0.4 μm) and are aligned in rows along the three ⟨001⟩ directions [42]. They were investigated here for two major reasons. Firstly, because of their ability to be cast as single grains that will be used to witness epitaxial grain growth during the HCPEB treatment. Secondly, because they are strengthened by coherent precipitates that were expected to witness the high temperature thermal cycles on heating (dissolution) [43]. It was also demonstrated recently that these coherent precipitates contained in superalloys do not induce the formation of a high density of craters under HCPEB [40]; craters that would potentially modify the local stress and, thereby, the solidification or solid state modifications on cooling [13].

2.2. HCPEB treatment

The HCPEB treatments were carried out using a “HOPE-1” source [16,30], a modified version of the Nadezhda-2 type source initially developed in Tomsk [3]. The following treatment parameters were used: energy densities of 4 J/cm^2 (for the Al alloy) or 6 J/cm^2 (for the superalloys) and a pulse duration of 1.5 μs. A number of 15 pulses (each separated by about 10 s) was used for the treatments. Small 10 × 10 × 8 mm specimens of the 2024 alloy were sampled from the sheet and the HCPEB treatment was applied with the electron beam parallel to the Normal direction of the sheet (perpendicular to the TD-RD plane). For the superalloys, the samples for HCPEB treatment were 2 mm-thick slices cut perpendicularly to the rod axis. Thus, the HCPEB treatment was carried out perpendicular to the rod axis with the electron beam directed along the [001] GD of the single grain. Before all the HCPEB treatments, the surfaces of the different samples were ground by using SiC sandpapers, followed by a polishing with diamond paste and finally with an OPS solution to ensure a high quality initial surface state (R_a about 0.05 μm).

2.3. Microstructure characterization

Surface and cross-sectional microstructures of the samples were observed by using a JEOL-7800F type SEM. The information about the microstructure and texture state of HCPEB-treated surface were also obtained by the JEOL-6500F type field emission gun scanning electron microscope (FEG-SEM), equipped with an electron back-scattering diffraction (EBSD) attachment (produced by Oxford). EBSD observations of the HCPEB treated sample surfaces were carried out without any further additional surface treatment. For cross sectional observations,