In situ study of the morphological changes on the surface of a duplex stainless steel during different heat treatments in inert atmosphere by means of high temperature laser scanning confocal microscopy

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Duplex stainless steels have a two-phase microstructure, in which ferrite and austenite are present in relatively large separate quantities and in approximately equal volume fractions. This kind of steel is widely employed in the chemical industry due to its good mechanical properties and corrosion resistance. These properties might deteriorate if stainless steels are heated improperly. During heat treatments new intermetallic phases may appear and the formation of new phases depletes the alloying elements in certain areas that lose their corrosion resistance [1-2]. Therefore, the rate and mechanism of solid state transformation in steels is of fundamental importance to avoid inadequate heat treatments that may lead to intergranular corrosion.

The aim of this work is to study in-situ the morphological changes that happen in a duplex stainless steel, Alloy 900 (EN 1.4462), during different heat treatments in inert atmosphere using a confocal laser scanning microscope (CLSM).

A "Linkam" heating stage can be accommodated in the stage of the CLSM "Olympus LEXT 3100" in order to observe the alloy evolution during the heat treatment. The sample is placed inside the ceramic sample cup. There is a gas-tight chamber for atmospheric control that allows carrying out heat treatments in an inert atmosphere. The maximum temperature that can be reached is 1500 °C, which is close to the melting point of different stainless steels. Prior to heat treatment, the specimen was wet abraded from 220 Silicon Carbide (SiC) grit to a 4000 SiC grit finish and polished with 1 micron alumina. Then, the specimen was put in the crucible and the stage was closed in order to maintain an inert atmosphere. Prior to heating, an argon flow passed through the heating stage in order to purge the oxygen; during the test this argon atmosphere was maintained. After that, a heating ramp was programmed from 25 °C to 750 °C, 850 °C and 950 °C, respectively. These temperatures were held for 1 hour and images of the surface evolution were obtained. It is expected that the migrating fronts may be observed as disruptions of the surface that occurs in the transformed regions.

Contrast changes observed by the CSLM system during solid phase transformation are a result of topographical changes caused by surface deformation due to the displacive and dilatometric nature of phase transformations. Initially, at 25 °C, the surface is homogeneous, without differences between the phases. When the temperature reaches 300 °C, both phases begin to differentiate; austenite has a higher thermal coefficient than ferrite, Thus, austenite grains grows quickly than ferrite grains. On the other hand, during the heat treatments of duplex stainless steels new phases (sigma and chi phases) appear and the percentage of one of the initial phases (ferrite) decreases [3, 4]. Then, when temperature reaches 600 °C some migration fronts begin to appear in the interface between ferrite and austenite grains. The height of these fronts grows with respect to the neighbour areas, being possible to measure this parameter with the CLSM software. Additionally these fronts become much wider with the duration of the heating. Finally, during the constant temperature heating the surface goes smoothing. Observations of these variations at 850 °C using the LSCM facility are shown in **Figure 1**. The evolution of the mean values of the height and width of these fronts during the heating at constant temperature (850 °C) is shown in **Figure 2**.

High temperature laser scanning microscopy provides an interesting tool for in-situ observations of phase transformations in stainless steels. The evolution of these transformations and the subsequent relationship with the corrosion resistance could contribute to a better selection of the heat treatment conditions [5].

References

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d) 800 °C

e) 850 °C, 5 minutes

f) 850 °C, 60 minutes

Figure 1. Confocal images of Alloy 900 surface obtained at different points of the heat treatment in inert argon atmosphere at 850 °C obtained by means of the CLSM



Figure 2. Evolution of the mean values of the height and width of the migration fronts during the heat treatment of Alloy 900 at 850 °C for one hour in innert atmosphere.