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Microstructural study of duplex stainless steels obtained by powder injection molding

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ABSTRACT

This experimental work is focused on the study of microstructural evolution during sintering of duplex stainless steels (DSS) obtained by powder injection molding (PIM). Ferritic 430L and austenitic 316L stainless steel powders were previously premixed in a 50/50 volume ratio and afterward they were sintered in low vacuum at different temperatures for 1 h. Microstructural analysis of sintered samples was conducted by means of scanning electron microscopy (SEM) and a compositional analysis of the alloying elements along different phases was performed by energy dispersive analysis of X-rays (EDS). Phase transformations were evaluated by X-ray diffraction (XRD) experiments, and the magnetic phase content was measured with a ferritoscope. The intensity of the main austenite diffraction peak decreases as sintering temperature increases to finally disappear in the sample sintered at 1100 °C. This destabilization of the austenite is probably related to a high Nickel diffusion detected from austenite to ferrite particles. Moreover, electron backscatter diffraction (EBSD) data were collected to quantify microstructural properties. Several EBSD pattern maps were acquired in order to define the amount of austenite phase. Due to the advantages of this technique a 0.5% of austenite could be detected after sintering at 1200 °C. After sintering process, the austenite content in sintered duplex stainless steels obtained through this processing route was lower than expected. Finally, Bain mechanism was proposed as an explanation to this phase transformation takes place. EBSD technique has been proved to be the most suitable to monitor the microstructure of sintered DSS.

1. Introduction

Duplex stainless steels (DSS) are characterized by a structure consisting of approximately equal amounts of ferrite and austenite. These steels combine some of the main characteristics of austenitic and ferritic steels [1]. The combination of these phases provides harder and more ductile steels than the austenitic and ferritic ones, respectively. The final composition and microstructure are obtained by interdiffusion of the alloying elements during sintering. Nowadays, there are several studies based on the development of duplex stainless steels by pressing powder metallurgy route [2–12]. Sintered DSS could be obtained through different methods: using prealloyed powders [3], mixing an austenitic powder with a ferritic stabilising element powder that during sintering will diffuse in the austenitic phase and will cause its destabilization producing the biphasic microstructure [4,5], or by premixing ferritic and austenitic powders in the adequate proportion [6–12]. In the last case, the selection of ferritic and austenitic starting powders ratio and sintering atmosphere obviously determine the microstructure obtained. Recently we have also reported the use of Powder Injection Moulding (PIM) technology to produce this kind of stainless steels. This technology normally use spherical particles, and it favours fundamental studies of sintering process, In these sense, the aim of this experimental work was to study the microstructural evolution during of premixed ferritic and austenitic starting powders processed by using spherical particles. In particular, the austenite–ferrite transformation during heating, associated Cr and Ni diffusion will be studied. To achieve this goal different characterization techniques were used, including Scanning Electron Microscopy combined with EDS microanalysis, and Electron backscatter diffraction (EBSD), X-ray Diffraction, as well as magnetic measurements employing a ferritoscope. As a result, a good understanding of the process would allow the obtaining of customized duplex stainless steels.

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2. Experimental

Gas atomized 316L and 430L stainless steel powders employed in this work were premixed in a 50/50 volume ratio. The chemical composition of these powders is summarized in Table 1. The d_{90} size-distribution parameter was 26 and 16 for the austenitic and ferritic powder, respectively.

The binder employed to prepare the feedstock was composed by high density polyethylene (HDPE) and paraffin wax (PW) in a volume ratio of 50/50. The experimental details of PIM process are given in Ref. [13]. Three-point bending parts were conformed to dimensions close to 63 × 12 × 3 mm. After debinding, the samples were sintered in low vacuum (<10^{-2} mbar) at different temperatures between 950 and 1300 °C for 1 h. The density of sintered parts was determined by Archimedeś method, and each value was calculated after testing 5 samples. The final carbon content was analysed by LECO instrument and all the samples presented values lower than 0.02%.

Sintered parts were examined in a Philips XL30 scanning electron microscope equipped with SE and BSE detectors. These samples were previously electrochemically etched with a KOH 3 M solution. Five micrographs of each sintering temperature were processed employing Image ProPlus 5.0 program with bright and contrast adjustments in order to quantify the ferritic phase content of the samples.

A compositional analysis of the alloying elements along different phases was performed by means of energy dispersive analysis of X-rays (EDS) employing EDAX software. All the microanalyses were performed under a voltage of 20 kV and a live time of 50 s.

Electron backscatter diffraction (EBSD) has been used extensively in both single phase metals and duplex stainless steels, to study grain structure, deformation and the initiation of recrystallisation [14–16]. Specimens for EBSD characterization were prepared by removing the surface layer by grinding and subsequently vibratory polishing with colloidal silica.

XRD patterns were recorded in a Philips X́Pert difractometer with Cu Kα radiation, a voltage of 40 kV and a current of 40 mA. The 2θ region analysed was 20–80° with a step scan of 0.02° and a counting time of 20 s per step.

Magnetic methods seem to be adequate for testing wrought duplex stainless steels, since they are sensitive to the amount and structure of the ferromagnetic ferrite phase. Magnetic saturation as well as the transition temperature was found to be proportional to the ferrite content in cold rolled duplex stainless steels [17]. The magnetic phase content was determined with a Fischer FMP30 ferritoscope and the mean values were reported after ten measurements (values higher than twice standard deviation were discarded).

3. Results and discussion

3.1. Density

Fig. 1 shows the relative density of the injection moulded duplex stainless steels parts sintered in low vacuum at different temperatures. The density increases gradually by increasing the sintering temperature up to 1250 °C where almost full density was achieved. From this temperature density values remain almost constant and close to 97% of the theoretical one. Moreover, Vickers hardness values increase from samples sintered at 950 °C till sintered at 1250 °C. After this sintering temperature, hardness remains almost constant at 280 HV30.

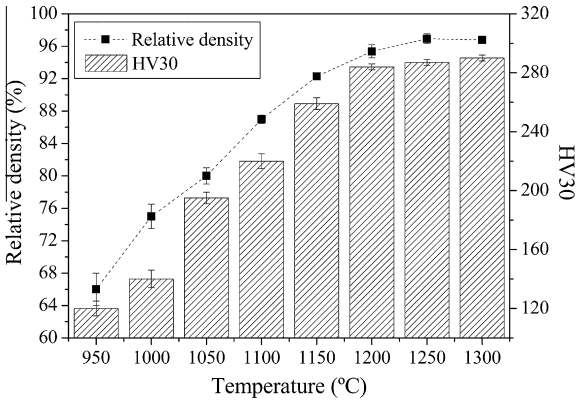

Fig. 1. Evolution of relative density and hardness of DSS sintered in low vacuum.

3.2. Scanning electron microscopy and EDS

Scanning electron micrographs of sintered samples between 950 and 1250 °C are shown in Fig. 2 (these micrographs were taken with BSE detector). A biphasic microstructure characteristic of these steels is found, which is constituted by dark regions distributed in a bright matrix. In principle, and taking into account the chemical analysis (see Fig. 3), dark and bright contrasts are assigned to ferrite and austenite phases, respectively. The residual porosity (black contrast), characteristic of PM steels, is also observed and it is reduced as the sintering temperature increases, in agreement with the density measurements.

At 950 °C the spherical morphology of the starting powders can be still observed and necks between particles indicate the beginning of sintering process (Fig. 2a). At this temperature spherical ferrite and austenite particles are clearly distinguished. As sintering temperature increases (for instance at 1100 °C (Fig. 2b)), neck growth is evident, especially in the bright particles, while dark regions distributed in a bright matrix can be also observed. At 1200 °C (Fig. 2c), spherical morphology of starting powder cannot be appreciated and when increasing temperature the coalescence of the ferritic regions can be detected. The percentage of ferrite (dark regions) in sintered sample at 1250 °C, as calculated by quantitative metallography of the micrographs, was close to 20%. However, this ferrite content is much lower than the ferritic stainless steel powder initially added (50 vol.%). As it can be seen grain size of ferrite phase is bigger after sintering at higher temperature.

It is worth to be mentioned, that in this kind of steels, usually a heat treatment together with a fast cooling is required after sintering to prevent precipitation of secondary phases [10,18]; however, in our case no evidence of the presence of these phases was found in any case.

Due to sintering process involves mass transfer, compositional analysis in two phases were performed by EDS. Ferrite stabilizing elements, as Chromium, are concentrated in ferritic areas and, austenitic stabilizing elements, as Nickel, in austenitic zones. In order to study the diffusion process, the evolution with sintering temperature of Cr and Ni amounts inside the grain in both phases was firstly analysed. These elements have been chosen because they are decisive for the stabilization of both ferrite and austenite phases. In Fig. 3 the amount of Cr and Ni, in dark and bright phases, are presented against sintering temperature.

As seen in Fig. 3a, the Cr content of ferrite phase (dark phase in SEM images) increases with respect to ferritic starting powder. It increases till 20.5 wt.% at the highest sintering temperature. However, the amount of this element in the austenite phase (bright

Table 1
Chemical composition of 316L and 430L gas-atomized powders provided by the supplier (wt.%).

316L								
Fe	Cr	Ni	Mo	Mn	Si	C	P	S
Balance	17.29	10.83	2.37	1.44	0.65	0.022	0.023	0.006

430L						
Fe	Cr	Mn	Si	C	P	S
Balance	16.20	0.71	0.75	0.026	0.029	0.008