



# Influence of sinter-cooling rate on the mechanical properties of powder metallurgy austenitic, ferritic, and duplex stainless steels sintered in vacuum

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## ABSTRACT

Austenitic, ferritic and duplex stainless steels obtained through powder metallurgy technology were sintered in vacuum. Powders were compacted at 650 or 750 MPa and sintered in vacuum with two sinter-cooling rates (furnace- and water-cooling). Mechanical properties, using tensile testing and hardness measurements were evaluated. A full microstructural study of the three types of stainless steels was performed. The mechanical behavior was a function of the sinter-cooling rate and the chemical composition. Duplex stainless steel showed the best mechanical behavior. The use of high compaction pressure and water-cooling process promoted the best mechanical results for all compositions.

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## 1. Introduction

Powder metallurgy (PM) stainless steel (SS) components constitute an important and growing segment of the PM industry. Austenitic grades have been widely used in automotive, marine, food, biomedical industries due to their high corrosion resistance [1]. However, ferritic SS have gained wide acceptance in automotive exhaust systems, containers and other functional applications owing to good fabrications at low cost, and their resistance to corrosion and oxidation [2–4]. Conventional (non-PM) duplex stainless steels have a combination of mechanical strength, toughness and corrosion resistance that make them attractive for numerous applications [5]. For duplex PM SS is also possible to achieve a high corrosion resistance [6].

PM stainless steels, as compared to their equivalent non porous materials, show restrictions in their applications due to the relatively poor mechanical and corrosion properties [7]. Therefore, there is always a thrust to improve such properties [8–10].

For PM austenitic SS, there were attempts to increase densification by supersolidus liquid phase for SS sintered in hydrogen atmosphere [11] or by increasing nitrogen sintering temperature to increase tensile and fatigue strengths [11] or, finally, by additions of elements such as silicon to promote the densification rate [12].

It has been reported some results on the nanoindentation

hardness of some PM duplex SS sintered in a vacuum and slow sinter-cooling [13]. It has been explained by the solid solution hardening in ferrite, because of the internal strain hardening between ferrite and austenite and because of the new inter-diffusion area at particles boundary [13]. Furthermore, the copper [14,15] and boron [16] additions, which activate sintering and enhance densification, closing the residual porosity and increasing ductility, improves the mechanical behavior.

PM austenitic and ferritic SS sintered in vacuum show a simple microstructure of austenite and ferrite respectively [17,18]. However, PM duplex SS shows complex microstructures that have been often analyzed by using the Schaffler's diagram. Non-conventional microstructural features such as the mixed constituent can be due to the combined content of alpha-genic and gamma-genic elements that at sintering temperatures show high diffusivity [16,19,20]. Another phenomenon found in PM duplex SS is related to the presence of sigma phase, carbides and other intermetallic compounds [21].

It is clear that the microstructure is influenced by the sinter-cooling rate. Austenitic and ferritic SS sintered in vacuum and after slow cooling during sintering (average cooling rate of 5 °C/min) showed the presence of brittle phases resulting in a decrease of the corrosion resistance [6]. Therefore, it is advisable to avoid these undesirable metallurgical transformations. Powder injection molded 316L, sintered in a vacuum with a cooling rate of 10 °C/min, showed higher mechanical properties and corrosion resistance than those cooled at 5 °C/min [22].

For PM duplex SS sintered in vacuum the effects of sinter-

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cooling rate on mechanical properties and corrosion resistance have been partially investigated. The “sinter-hardening” proposed by Dobrzanski et al. [23,24] and cooling with N<sub>2</sub>, proven its advantage for corrosion properties but the effect on mechanical behavior was not investigated.

Previous works of the authors [25,26] report that PM SS sintered in nitrogen have proven that nitrogen absorption causes the formation of chromium nitride precipitates, which reduces ductility and promotes chromium depleted areas. The dissolution of these secondary phases by a post-sintered solution annealing is possible. Water quenching from sintering temperature is another possible way to avoid these brittle precipitates [27,28]. Mechanical properties of PM SS sintered in nitrogen has been previously published by the authors [28].

The main issue of the present study is to investigate the microstructure and mechanical properties of stainless steels sintered in vacuum and sinter-cooled at slow and high rates. Three typical PM SS has been chosen, specifically a ferritic 430L, an austenitic 316L and a duplex 50% ferrite powder and 50% austenite powder.

## 2. Experimental

Two prealloyed and water atomized powders were used as raw materials: AISI 430L (0.018 wt% C, 1.16 wt% Si, 0.18 wt% Mn, 16.9 wt% Cr, 0.10 wt% Ni, Fe bal.) and AISI 316L (0.021 wt% C, 0.87 wt% Si, 0.20 wt% Mn, 16.1 wt% Cr, 13.55 wt% Ni, 2.24 wt% Mo, 0.02 wt% Cu, 0.1%V, Fe bal.). The powder characteristics of AISI 430L were: apparent density 3.0 g/cm<sup>3</sup>, flow rate 26 s/50 g and nominal particle size < 50 μm. The powder characteristics of AISI 316L were: apparent density 3.1 g/cm<sup>3</sup>, flow rate 25 s/50 g and nominal particle size < 150 μm. The duplex SS was obtained by premixes of 316L and 430L prepared in a turbular mixer. The content of AISI 316L/430L was set to 50/50 wt%. This material was designated as 50DSS and the two simple materials (430L and 316L) were labeled as base materials.

Dog-bone tensile test specimens [29] were uniaxially compacted using a floating die at selected compaction pressures for selected compaction times. Zinc stearate was used as die lubricant. Compactions at 650 and 750 MPa for 300 s were chosen. Sintering in vacuum (11 Pa) at 1250 °C for 1 h was set. It was found by chemical analysis that there were no chromium losses. After sintering, two different sinter-cooling processes were applied. Some samples were cooled at a slow rate of 5 °C/min in the sintering furnace (referred to from now on as “furnace-cooling” process). Some others were submitted to fast cooling by direct immersion in water (designated from now on as “water-cooling” process). The specimens were referred to as “furnace-cooled” and “water-cooled” samples, respectively.

Image analysis was used to study the porosity of polished samples. Seven fields were taken per sample. Images were digitized and calibrated. The pores were identified as the black pixels and they were computed to calculate the area of each pore.

Samples were polished and etched before observation by optical metallography. The electrochemical etching with oxalic acid (ASTM A262, Practice A [30]) helped to define the location of the chromium precipitates. X-ray diffraction (XRD) was used to identify some of the phases. Finally, the samples were etched with Vilella’s reagent for their analysis by scanning electron microscopy with energy dispersive analysis of X-rays (SEM/EDS).

Tensile tests were performed following the ISO 6892-1 standard [31]. Tensile strength and ductility were evaluated. The apparent hardness (i.e. the value obtained when indentation is the result of plastic deformation of the material and the pores) was evaluated by the Vickers method, using a load of 30 kp (294 N) during 30 s. An average value of five indentations was given as the hardness value.

**Table 1**  
Porosity values for 316L, 430L and 50DSS.

Sample	Compaction pressure (MPa)	Furnace-cooling Porosity (%)	Water-cooling Porosity (%)
316L	750	13.95	14.04
	650	14.96	14.36
430L	750	11.33	10.24
	650	12.10	11.50
50DSS	750	14.2	13.72
	650	15.20	14.83

## 3. Results

### 3.1. Porosity and microstructural characterization

The porosity results are collected in Table 1. As expected the higher the compaction pressure the lower the porosity. No significant change in porosity was found from furnace- and water-cooled samples. It can be pointed out that the water-cooled samples show similar or lower porosity than the furnace-cooled specimens.

The microstructure, as expected, is a function of the chemical composition of the samples and the sinter-cooling rate. First, the microstructures of the furnace-cooled samples are discussed and second, the effect of water-cooling is commented. The microstructures are respectively collected in Figs. 1 and 2 for the compaction pressure of 650 MPa. A higher pressure (750 MPa) did not affect to the microstructures; the only difference was a lower degree of porosity (Table 1).

A typical austenitic microstructure with mainly transgranular (TG) precipitates was observed for furnace-cooled 316L (Fig. 1a). For the furnace-cooled 430L (Fig. 1b) a typical ferrite phase with slight intergranular (IG) and TG precipitations were observed. The furnace-cooled 50DSS revealed a more complex microstructure (Fig. 1c), which differed from the austeno-ferritic biphasic structure observed in conventional duplex SS. Austenite and ferrite grains were identified but also a mixed constituent [25], which is in this case a mixture of ferrite and austenite was observed. This mixed constituent was hard to distinguish by conventional optical microscopy and will be latter discussed. In Fig. 1c it can be seen austenite grains with some TG precipitation, ferrite islands and dark areas corresponding to the mixed constituent.

The microstructure of water-cooled 316L was entirely austenitic with a slightly lower grain size than the furnace-cooled 316L (Fig. 2a). There was also a lower amount of precipitation. For water-cooled 430L a higher quantity of precipitates than for 316L was observed (Fig. 2b). Comparing austenitic and ferritic samples, the effect of water-cooling is different, while for 316L the tendency is to obtain a clean microstructure of austenite grains, the contrary is observed for 430L.

For water-cooled 50DSS, the same three constituents as for furnace-cooled was observed but the ratio changed (Fig. 2c). The constituents were: the mixed constituent [25], but in this case is rich in martensite, the austenite grains and some ferrite islands that were in lower amount than for the furnace-cooled. The mixed constituent showed some isolated TG precipitates inside.

Optical microscopy observation is not enough for the full identification of the microstructures, therefore additional techniques such as XRD and SEM/EDS were used. The XRD patterns of all the samples are shown in Fig. 3. The peaks of the austenite phase for 316L were predominant for the two sinter-cooling rate. However, peaks identified as ferrite/martensite phase were also observed and were more significant for the furnace-cooled sample. It is relevant to remind that ferrite and martensite phases have the same peaks in the diffractograms, therefore there is no way to

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