INFLUENCE OF DELTA FERRITE ON MECHANICAL PROPERTIES OF STAINLESS STEEL PRODUCED BY MIM

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Abstract: *Parts produced by Metal Injection Molding of stainless steel powders are sintered at very high temperature, usually above 1300°C, to obtain full density. Under these conditions, a certain amount of delta ferrite is stabilized in the as sintered microstructure. Delta ferrite enhances sintering, since volume diffusion in the bcc lattice is faster than in the fcc austenite lattice. However, when maintained in the as sintered microstructure, it tends to influence the mechanical properties.*
The effect of the amount of delta ferrite on the tensile strength and the fatigue strength of an austenitic stainless steel produced by MIM was studied in the present work. The content of delta ferrite was varied by changing the sintering temperature and the results of the mechanical tests were correlated to the microstructural features of the materials.

Keywords: *MIM, 316L stainless steel, delta ferrite, mechanical properties.*

1. INTRODUCTION

Metal Injection Molding (MIM) is a net shape technology which uses powders as raw material to produce parts characterized by a geometrical complexity with close dimensional tolerances (German 1997). It has been intensively developed in the last three-four decades, and finds application in different fields as, for instance, mechanical, automotive, biomedical, hobby and sport equipments industry. The powders are mixed to a binder to obtain a feedstock having the proper rheological properties to be injected in a die cavity and to keep the shape once the injection pressure has been removed. The green parts are then extracted from the die and treated to eliminate the binder. The debinded parts, called brown, are then sintered at high temperature to reach a final density very close to the theoretical one.

MIM is increasingly used to produce parts for biomedical industry. As an example, implantable prosthesis and external devices are produced by stainless steel, in particular the austenitic (Kyogoku *et al.*, 2000 and Loh *et al.*, 1996) and the precipitation hardening (Muterlle *et al.*, 2008 and Wu *et al.*, 2002) grades. In the case of austenitic stainless steel, since sintering is carried out at very high temperature (above 1300 °C) the final microstructure may contain delta ferrite (Collins, 2002). This constituent is expected to influence the mechanical and corrosion resistance to some extent.

In this work, the effect of delta ferrite on the tensile and fatigue resistance of an AISI 316L stainless steel was investigated. Different amounts of delta ferrite were obtained by changing the sintering temperature and by heat treatment. The results of the mechanical tests were interpreted on the basis of the microstructural characteristics and correlated to the fracture morphology investigated at the Scanning Electron Microscope (SEM).

2. EXPERIMENTAL PROCEDURE

A pre-alloyed gas atomized 316L powder was mixed with a proprietary binder and feedstocks were molded to produce the specimens for tensile tests according to ASTM E 8M-03 – Standard Flat Unmachined Tension Test Specimen for Powder Metallurgy (P/M) Products. The same specimens were produce for fatigue tests, as well. Debinding was carried out in two steps: dissolution in water, to eliminate 80% of the binder, followed by thermal decomposition.

The samples were sintered in TAV (Caravaggio, Italy) vacuum furnaces with 1 hour isothermal holding in 100 mbar Ar backfilling at the temperatures reported in Table 1.

Table 1. Sintering conditions.

Materials	Sintering Conditions
316L NF	Sintering at **1360°C** in graphitic furnace
316L F	Sintering at **1380°C** in metallic furnace

Cooling from the sintering temperature was carried out with 1 bar nitrogen flux. A third material was produced carrying out a heat treatment at 1390°C for one hour on 316L F; it is called **316L HF** in the following.

The carbon analysis was carried out by LECO CS125. Density was measured by the water displacement method.

The microstructure was investigated by Light Optical Microscopy (LOM) after etching with a 25% distilled water, 50% HCl and 25% HNO_3 solution. For the quantitative determination of the amount of delta ferrite, specimens were etched with a 5ml distilled water, 2.5g Potassium hydroxide and 2.5g Potassium ferrocyanide solution, and characterized by Image Analysis.

Microhardness (HV0,02) and hardness (HV10) were measured.

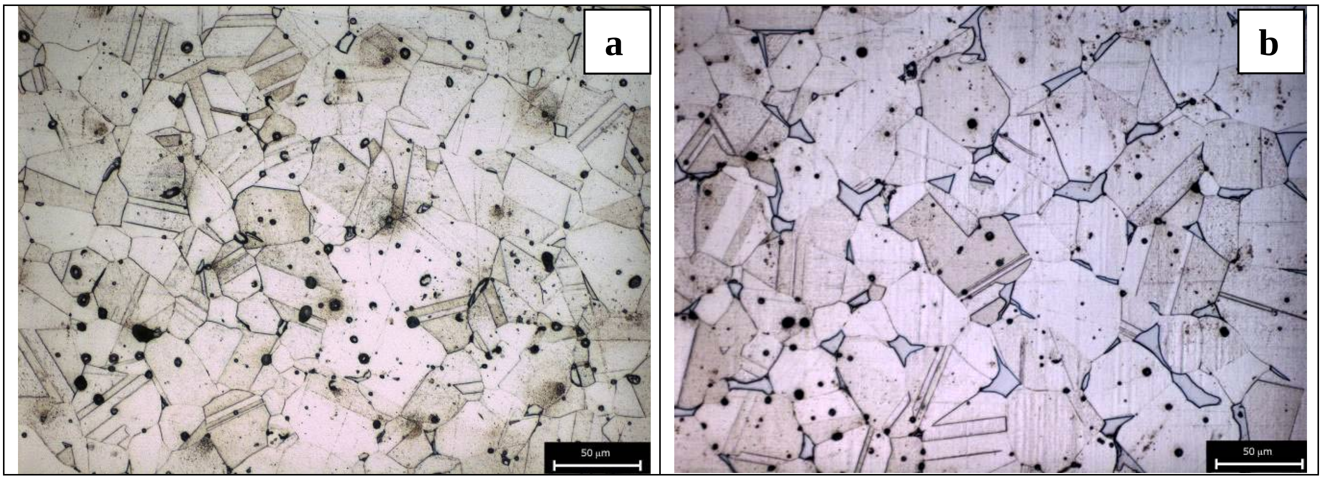
Tensile tests were carried out on an Instron machine with a strain rate of 0.2 s^{-1} and measuring strain with an axial extensometer with a gauge length of 12,5 mm.

High cycle fatigue tests were carried out on a Rumul Mikrotron 20kN machine with a frequency of 150Hz and a load ratio equal 0 (R=0). It was assumed $2x10^6$ cycles as run-out test. The fracture surfaces were investigated by SEM.

3. RESULTS AND DISCUSSION

3.1. Microstructure

Figure 1 show examples of the microstructure of the three materials investigated: NF (1a), F (1b) and HF (1c). The residual porosity is very low, and made of small, spheroidized and homogeneously distributed pores. Austenitic grains show several annealing twins, and the delta ferrite islands are isolated and distributed quite homogeneously, too. Figure 1d shows the effect of the specific etchant used to prepare the specimens for the quantitative determination of the amount of delta ferrite. Etching is localized at the austenite-ferrite interface, and the contrast between the two constituents is very sharp.



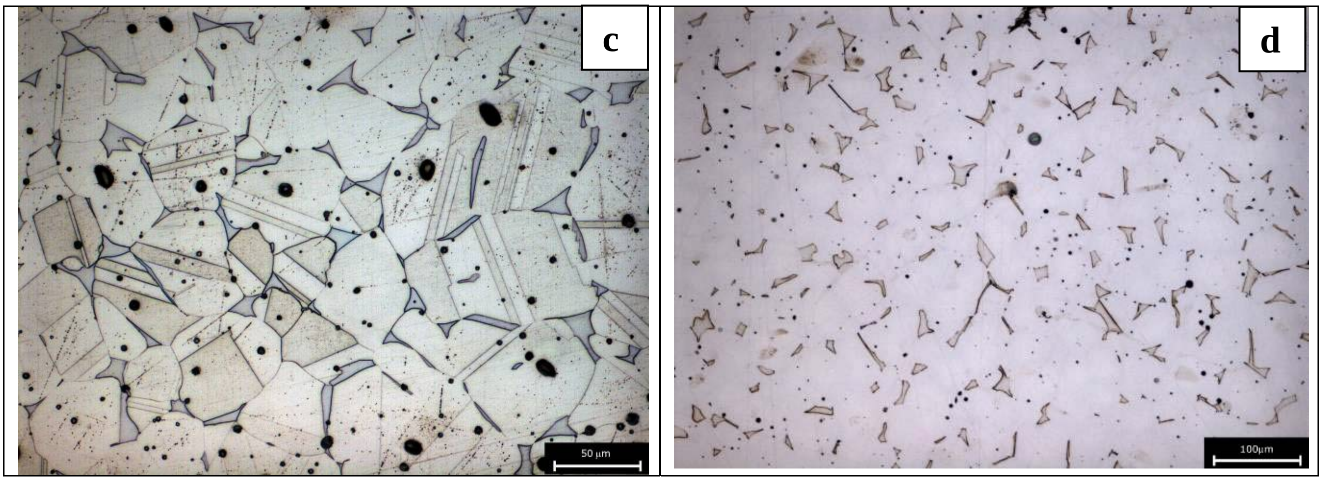


Figure 1. Optical micrographs of 316L materials: NF (a), F (b), HF (c) and after specific etching for the quantitative determination of delta ferrite by Image Analysis (d).

Table 2 reports the volumetric percent of delta ferrite, along with density and carbon content. On increasing the sintering temperature, delta ferrite content increases up to around 4.5%, and heat treatment leads to a further increase up to 8%. Density of the materials increases with the sintering temperature, as expected. At 1380°C, the material is practically full dense. Heat treatment does not change density. The carbon content is higher in the material sintered at the lower temperature, mainly because of the graphite heating bars which tend to reduce decarburization.

Table 2. Chemical analyses and ferrite composition.

Material	**delta ferrite, %**	**Density, (g/cm^3)**	**C, %**
316L NF	< 1	7.83±0.03 (98.5% of theor.)	0.0132
316L F	4.5±0.9	7.94±0.01 (99.8% of theor.)	0.003
316L HF	8±1	7.94±0.01 (99.8% of theor.)	0.003

3.2. Microhardness and hardness

Microhardness and hardness data is reported in table 3. Delta ferrite is harder than austenite, and on increasing its content in the material, hardness increases. Differences are rather low but significant.

Table 3. Microhardness and hardness of the investigated materials.

Samples		**Microhardness (HV0.02)**	**Hardness (HV10)**
316L NF	ferrite	Not measurable	111.1±3.1
	austenite	170.3±12.9	
316L F	ferrite	223.5±5.8	120.4±1.7
	austenite	163.5±1.2	
316L HF	ferrite	223.0±29	126.0±3.1
	austenite	167.5±11	

3.3. Mechanical properties

Figure 2 shows the tensile stress-strain curves of NF and F. The materials display a uniform plastic deformation up to the maximum stress, without any appreciable non uniform deformation.

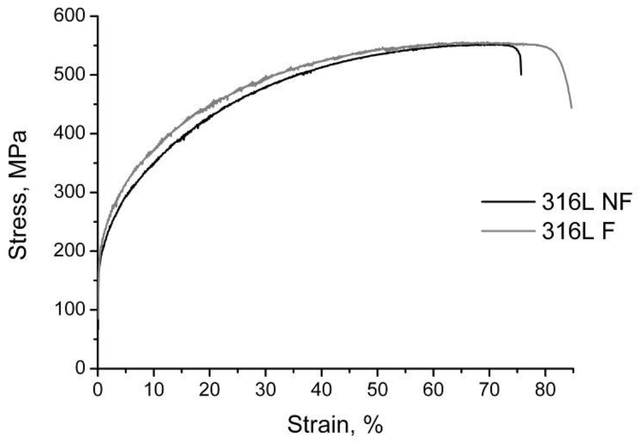


Figure 2. Tensile test curves for 316L materials

Table 4 reports the tensile properties of the three materials investigated. Results are comparable to those reported in the literature (Heaney *et al.*, 2004).

Table 4. Tensile properties of the investigated materials.

Samples	**Yield Strength (MPa)**	**Ultimate Tensile Strength (MPa)**	**Elongation %**
316L NF	186±1.5	549.9±3.9	65.7±9.3
316L F	194.1±5	555.4±0.5	82.7±2.6
316L HF	211.2±2	571.8±7.5	77.7±3

The increase in the sintering temperature from 1360°C to 1380°C causes an increase in both strength and ductility. This is due to the enhanced densification, but even to the increase in the ferrite content. This constituent is expected to increase strength, as actually observed. It also should cause a decrease of ductility, but the increase in density has a prevailing effect. Heat treatment increases strength but decreases ductility. This is only due to the increase in the ferrite content, since density does not change with heat treatment.

The fracture morphology is ductile, characterized by dimples as showed in figure 3, relevant to F and significant of the other materials shows. The fracture morphology does not show any feature attributable to delta ferrite, since this constituent has a typical ductile behavior as well.

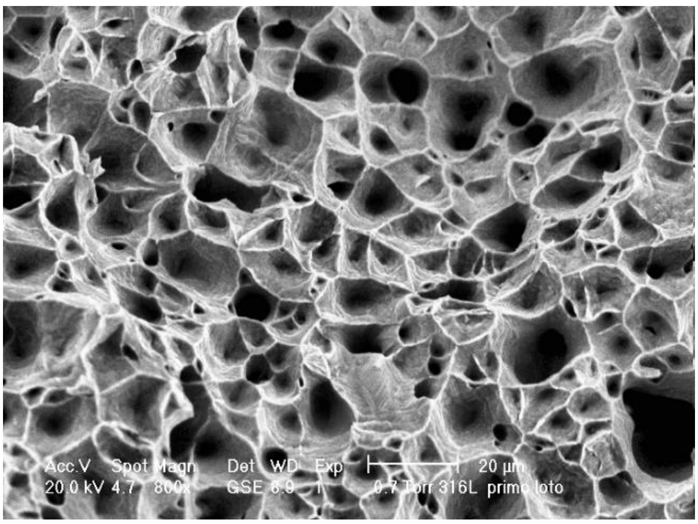


Figure 3. Tensile fracture surface of 316L F material

Table 5 reports the results of fatigue tests. The fatigue strength (FS) at $2x10^6$ cycles is reported, along the ratio between FS and UTS. Results are comparable to those reported in the literature (Kyogoku *et al.*, 2000).

Table 5. Fatigue tests.

Samples	Fatigue Strength 50% (MPa)	FS / UTS
316L NF	248±15	0.45
316L F	293±1	0.53
316L HF	272±16	0.48

Fatigue strength increases on increasing the sintering temperature (F versus NF), because of the increased density, strength and ductility. Here, the effect of delta ferrite is overshadowed by the enhanced densification provided by the higher sintering temperature. Fatigue strength is around one half of UTS. The effect of delta ferrite is shown by the comparison between HF and F. Here, fatigue strength decreases slightly despite the increased tensile strength, because of the decreased ductility. The FS/UTS ratio decreases, correspondingly.

The analysis of the fracture surface allows the nucleation of the fatigue crack to be individuated. It occurs on the surface, in correspondence of a pore, as figure 4 shows. There is an extensive slow propagation step of the fatigue crack (figure 5), followed by fast propagation by overloading with the typical ductile morphology (figure 6).

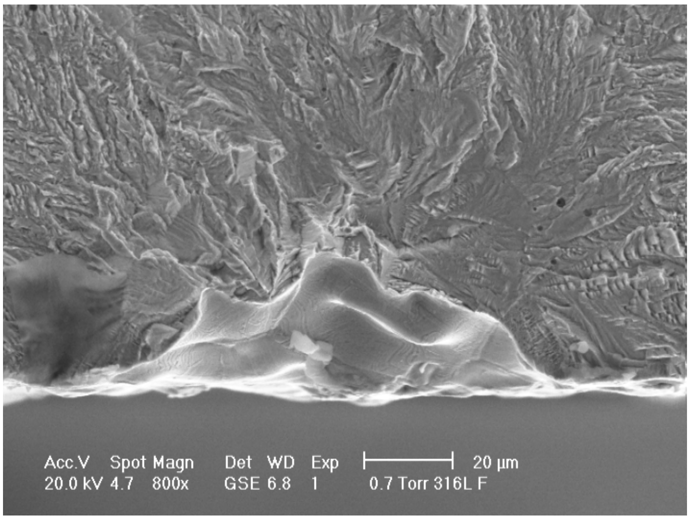


Figure 4. Nucleation site of the fatigue crack in 316L NF material

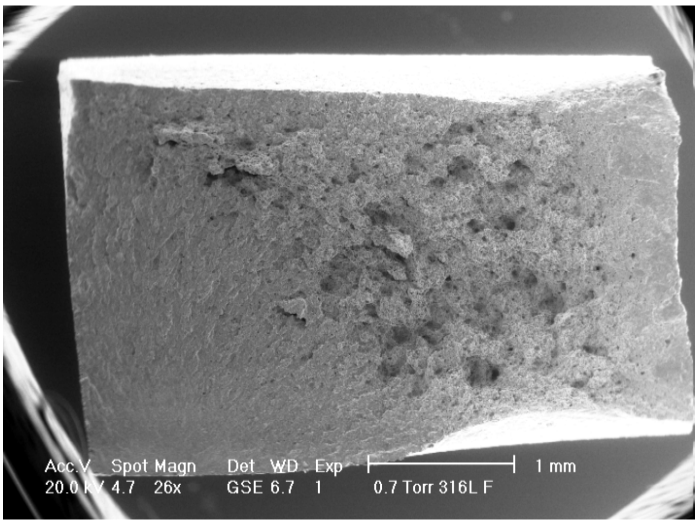


Figure 5. Fatigue fracture surface of 316L NF material

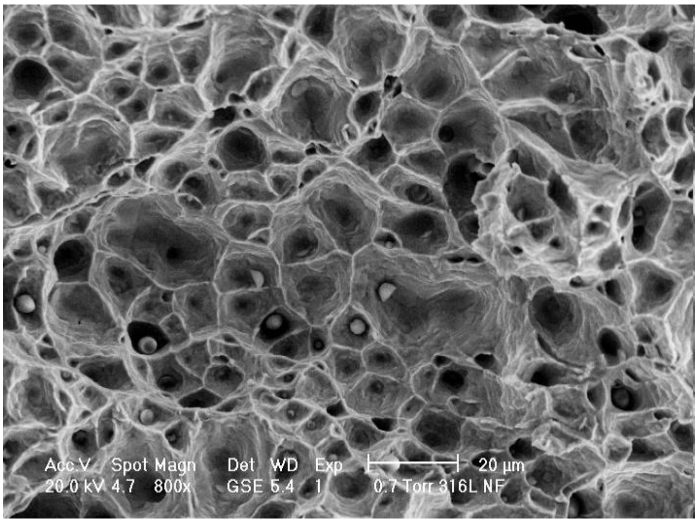


Figure 6. Ductile morphology of the fast fatigue crack propagation

As for tensile tests, there isn’t any appreciable effect of the presence of ferrite on the fracture surface. The analysis of the propagating crack confirms the absence of any preferential propagation path. The fatigue crack propagates through austenite grains as well as through delta ferrite grains.

4. CONCLUSIONS

Austenitic stainless steel tensile and fatigue specimens were produced by MIM to study the effect of the content of delta ferrite on mechanical properties. Delta ferrite content increases with the sintering temperature, and with a post-sintering heat treatment at high temperature. Sintering temperature increases density from 98.5% to 99.8% of the theoretical one.

Delta ferrite increases hardness and both yield strength and Ultimate Tensile Strength, correspondingly. On the other side, ductility decreases. This effect can be overshadowed by density when the increase in delta ferrite is due to an increase in the sintering temperature. In this case, the effect of density on ductility prevails on that of delta ferrite. As far as fatigue strength is concerned, it is correlated to tensile ductility; it decreases with the increase in the delta ferrite content at a constant density. No features attributable to delta ferrite were observed on the fracture surfaces.

5. REFERENCES

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