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Overview of Properties, Features and Developments of PM HIP 316L and 316LN

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Abstract. PM HIP 316L is an alloy that is of increased interest for nuclear applications since its recent ASME code case approval. Over the years, comprehensive data and understanding of the properties and features have been collected and evaluated which will be summarized in this article. Since the early developments of the PM HIP technology it has been observed that PM HIP alloys generally exhibit higher yield strengths compared to their conventional counterparts, a feature that applies well for 316L/LN. In this article this is demonstrated, both by using the Hall-Petch relationship as well as Pickering´s and Irvine´s empirically derived relationship between composition and grain size for austenitic stainless steels. Furthermore, a mechanism generating the increased yield strength in PM HIP vs conventionally manufactured 316L and 316LN will be proposed. Results also show that low oxygen contents itself is not a guarantee for good or increased performance in form of mechanical properties, but that there are other features that is of similar or perhaps even higher importance in order to achieve good properties. The results of this article include microstructural properties derived from EBSD measurements as well as tensile and impact properties in a wide range of test temperatures of PM HIP 316L and 316LN from several powder batches manufactured at different locations and processed with various HIP and heat treatment procedures. Finally, some results regarding creep properties of PM HIP 316L is presented.

Introduction

Austenitic stainless steel 316L is one of the most commonly known and used stainless steel grades and the performance and properties of this alloy in different product forms is well known. Powder Metallurgical manufacturing via Gas Atomization and Hot Isostatic Pressing is a manufacturing technology known to generate isotropic microstructures, high cleanliness and often improved mechanical properties. In light of the recent ASME code case approval for PM HIP 316L [1], the properties of this alloy via this manufacturing process has become of increasing interest [2-4]. This article will give an overview of the properties of PM HIP 316L/316LN, how properties can be affected by varying manufacturing process parameters and compare how they differ from the conventionally manufactured counterparts.

Microstructure

One of the large benefits with PM HIP manufacturing is that the microstructures of the manufactured components are homogeneous, isotropic and have high cleanliness. All these features apply also for PM HIP 316L/316LN and translates into excellent ultrasonic inspectability [4]. Regarding cleanliness, the clear majority of the non-metallic inclusions found in PM HIP 316L/316LN are well below 2.8 µm in size and are predominately constituted by oxides [2,3]. The oxides can originate either from the melt which are later trapped within the powder particles (bulk oxides) or from the surface oxide layer and oxide particles formed on each powder particle surface after solidification (surface oxides) [3,5]. The latter are often referred to as PPB (Prior Particle Boundary) inclusions in the HIPed microstructure and can form a network that affect ductility and toughness adversely if they are present in large amounts. The general perception from manufacturing experience is that formation of detrimental PPB inclusion networks is not an issue for PM HIP 316L/316LN if properly processed during manufacturing. Inclusions in PM HIP 316L/316LN have been observed to pin grain boundaries and affect grain size [4]. In Fig. 1 a general microstructure displaying grain size and grain orientation derived from EBSD (a) and a SEM image of small inclusions at high magnification can be seen (b) [2, 3]. In Fig. 2 the size distribution of non-metallic inclusions in different batches of PM HIP 316L and 316LN as well as conventionally manufactured 316L (hot rolled Ø50 mm bar) can be observed [2, 3].

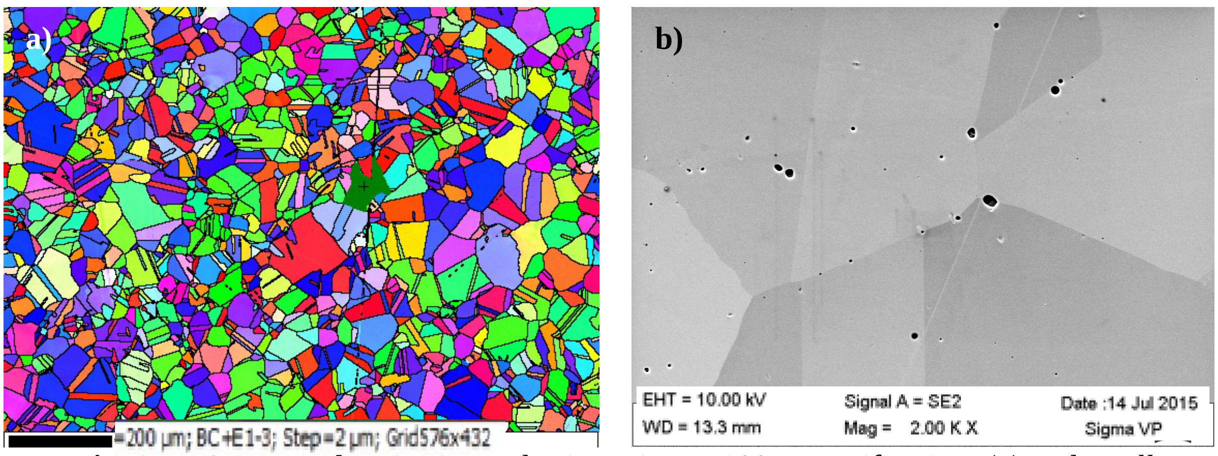

Fig. 1. *EBSD map of grain size and orientation at 100x magnification (a) and small inclusions some pinning grain boundaries, at 2000x magnification (b) in PM HIP 316L [2, 3]*

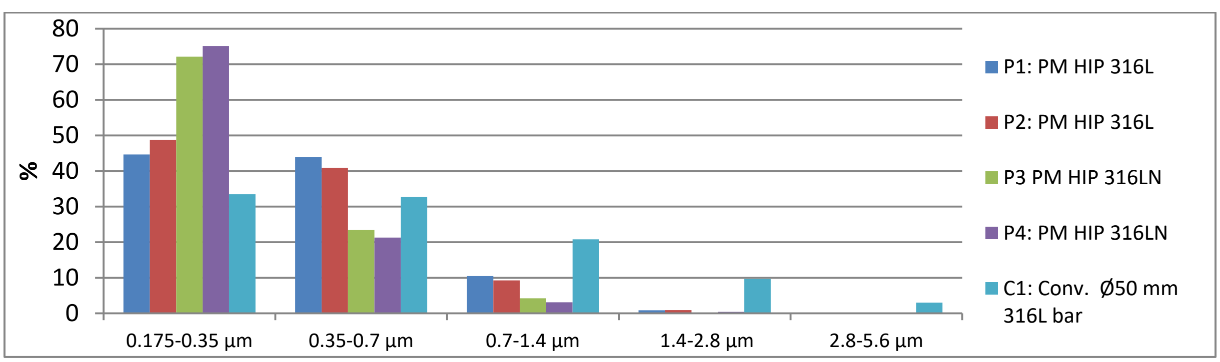

Fig. 2. *Size distribution of inclusions in PM HIP & conventional 316L/316LN [2, 3].*

Mechanical properties

Table 1 shows room temperature mechanical properties for PM HIP 316L components with wall thicknesses 60 - 600 mm and weights 10 - 3000 kg. All samples were HIPed at 1150ºC/3 h/100 MPa and heat treated at 1050ºC for 1.5 - 8 hours followed by water quenching.

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Table 1. *Average mech. properties ± st. deviation at room temperature for PM HIP 316L.*

Samples [#]	Rp0.2 [MPa]	UTS [MPa]	A [%]	Impact Toughness [J]
37	275 ± 13	582 ± 20	60 ± 2	204 ± 23

Looking at the tensile properties of PM HIP 316L and 316LN [2, 3], it appears as if the yield strength is generally higher than for conventionally manufactured counterparts [2, 8-10]. Pickering and Irvine et. al. derived an empirical relationship between composition and microstructural parameters and yield strength for austenitic stainless steels; Eq. 1 [6,7].

$$\mathrm{Rp0.2\ (MPa)} = 15.4[4.4+23(\mathrm{C})+1.3(\mathrm{Si})+0.24(\mathrm{Cr})+0.94(\mathrm{Mo})+1.2(\mathrm{V})+0.29(\mathrm{W})+2.6(\mathrm{Nb})+1.7(\mathrm{Ti})+0.82(\mathrm{Al})+32(\mathrm{N})+0.16(\text{delta ferrite})+0.46d^{-0,5}]. \quad (1)$$

In Eq. 1 elements are in weight percent, delta ferrite in percent and *d* is the linear intercept grain size in millimeters. Fig. 3 displays both measured and predicted yield strength at room temperature per Pickering and Irvine et. al. of different PM HIP 316L and 316LN batches as well as for conventionally manufactured counterparts found in literature [8-10]. P4 is a PM HIP 316LN sample characterized identically as P3. As can be observed the Pickering-Irvine prediction is relatively accurate for conventional 316L while the yield strength of the PM HIP samples is consistently underestimated. This is an indication that the PM HIP samples exhibit a strengthening contribution from other factors than composition, delta ferrite and grain size.

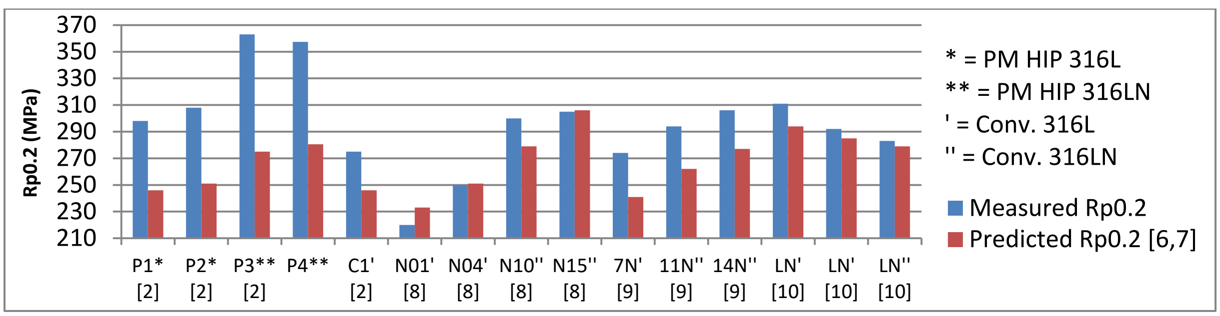

Fig. 3. *Measured and predicted yield strength per Pickering and Irvine [6,7] at room temp.*

The main strengthening contributors per to Eq. 1 is grain size, N and C content. If the strengthening contribution from these factors are subtracted from the measured yield strength, a theoretical yield strength (denoted as Rp0.2-n) normalized with regard to these parameters should be obtained. Fig. 4 shows a plot of Rp0.2-n versus amount of oxygen containing inclusions >0.175 µm per mm^2. As can be observed there is a good correlation between these parameters, indicating a strengthening effect from the oxygen containing inclusions in PM HIP 316L/316LN which is not present in the conventionally manufactured 316L.

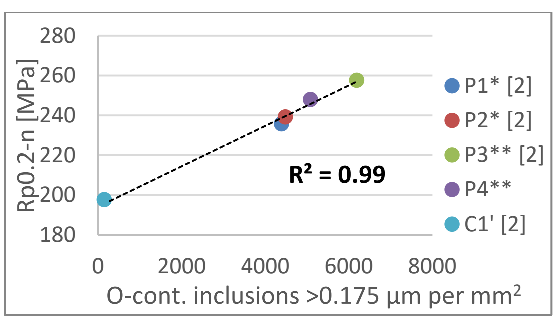

Fig. 4. *Rp0.2-n vs. O-cont. incl. per mm^2*

In eight different PM HIP 316LN samples of similar composition, the grain size was measured with a HKL Nordlys EBSD and the amount of inclusions >0.175 µm were measured using automated SEM-EDS with a Zeiss Sigma FEG-SEM

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using the Aztec and Inca softwares provided by Oxford Instruments. According to the Hall-Petch relationship shown in Eq. 2 there should be a linear correlation between yield strength and grain size [11,12]. However, this correlation was relatively poor for these samples as can be observed in Fig. 5.

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}} \tag{2}$$

In Eq. 2 σ_y is the yield strength, σ_0 is the intrinsic yield strength (i.e. the yield strength of the material with infinitely large grain size, also called internal friction stress), k_y is a material specific constant, and d is the mean intercept grain size. The yield strength contribution from the grain size of the samples were calculated in which k_y was chosen to be 164 MPa·μm$^{-0.5}$ as derived for 316L [13]. The calculated grain size contribution was then subtracted from the measured yield strength for each sample to obtain the intrinsic yield strength/internal friction stress, σ_0. In Fig. 6 the intrinsic yield strength/internal friction stress σ_0 is plotted versus the ECD (Equivalent Circle Diameter) of oxides larger than 0.175 μm. As can be observed there is a good correlation between these parameters, indicating a strengthening effect from the oxide inclusions which could account for yield strength variations aside from grain size.

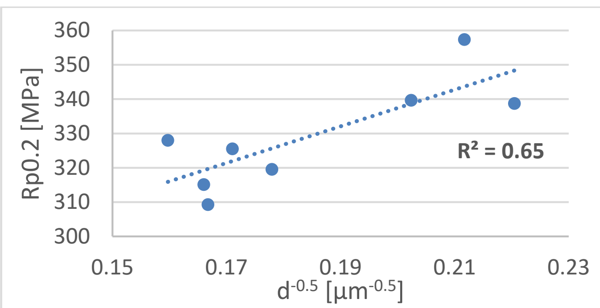

Fig. 5. *Yield strength vs. inverted square root of the mean intercept grain size.*

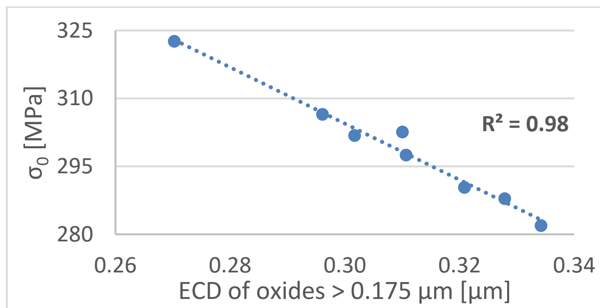

Fig. 6. *Intrinsic yield strength σ_0 vs oxygen containing inclusions >0.175 µm per mm^2.*

In both cases where PM HIP 316L/316LN samples were compared with conventional counterparts utilizing Irvine and Pickering´s relationship and where eight PM HIP 316LN samples were compared with the Hall-Petch relationship, results indicate a strengthening mechanism by oxygen-containing inclusions in the PM HIP samples. A general feature of PM HIP alloys is that they generally exhibit higher yield strength compared to conventional counterparts, a feature that is valid for PM HIP 316L and 316LN. A possible explanation for this could be that relatively large amounts of small oxygen containing inclusions act as small precipitates impeding dislocation movements during tensile strain, i. e. Orowan strengthening. Such strengthening effect should be stronger for oxygen containing inclusions smaller than 0.175 µm, but these are more difficult to characterise qualitatively and quantitatively. This theory is strengthened by the observation that the yield strength is reduced by 6-7 % in PM HIP 316LN when the oxygen content is reduced by 47 - 55% [14]. The samples with lower yield strength exhibited larger grain size, but utilizing Eq. 1 and 2. of this study it can be estimated that this can only account for a small amount of the yield strength reduction.

The impact toughness of PM HIP materials is a topic often discussed. Recently there has been raised concern as to why PM HIP 316L seems to drop in impact toughness at cryogenic temperatures [2,3,15]. It appears as if this decrease in impact toughness at cryogenic

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temperatures is caused by the relatively large amounts of small inclusions. Inclusions also affect impact toughness at room temperature, but due to the strength increase and ductility decrease of the matrix at lower temperatures the inclusions become more detrimental [3]. The impact toughness for several batches of PM HIP 316L/316LN and conventional 316L from different manufacturers at temperatures -196°C – 300°C can be seen in Fig. 7. As can be observed the impact toughness drops at -100°C and -196°C for the PM HIP samples while this is not the case for the forged 316L. Another observation is that the PM HIP materials can meet and exceed the conventional materials at and above room temperature.

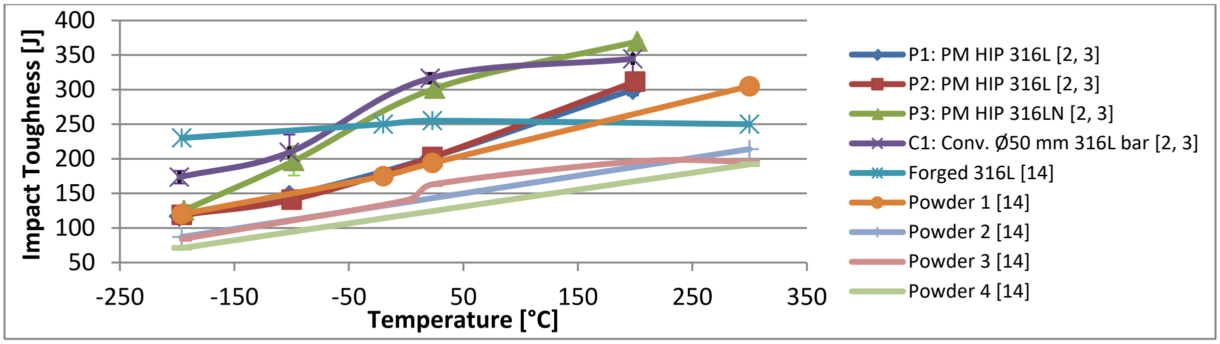

***Fig. 7.** Impact toughness for PM HIP and conventional 316L/LN between -196 to 300°C.*

In a similar study for PM HIP 316LN the impact toughness at -196°C increased by ~260 % (from 93 to 243 J), highlighting that oxides are a larger issue at cryogenic temperatures [17]. However, the total oxygen content is not a conclusive indicator on how the materials will perform regarding impact toughness. As explained previously, the total oxygen content in PM HIP materials originate both from bulk oxides and surface oxides [3,5]. The latter source of oxygen has a more detrimental effect on impact toughness as it is known to form a network of oxides on the PPBs if the surface oxygen content is high. PM HIP 316L/LN is known to have a ductile fracture, and voids are normally nucleated around inclusions during deformation [2]. This has been observed in in-situ SEM tensile testing studies of PM HIP 316L of which an example can be seen in Fig. 8. These voids grow during further deformation and ultimately coalesce with each other leading to fracture [2,3,18]. Having larger amounts of oxides in the microstructure as an effect of higher oxygen content will result in increased number of sites for void nucleation and reduced space for voids to grow without coalescing with adjacent voids, thus accelerating the fracture propagation. In the case of PPB oxides, void coalescence occurs almost immediately after void nucleation due to the vicinity of each PPB oxide leading to significantly reduced impact toughness.

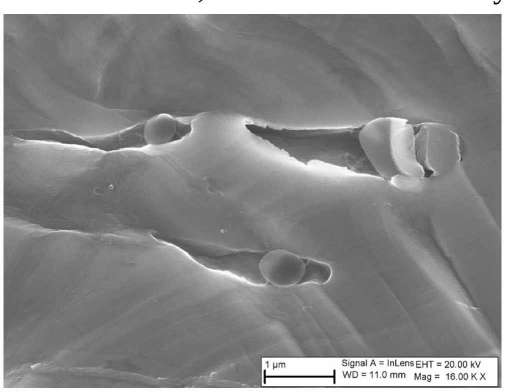

***Fig. 8.** In-situ SEM tensile test showing void nucleation and growth around inclusions for PM HIP 316L at 16000x magnification [2].*

Fig. 9 shows an example of how the impact toughness can vary between different samples of PM HIP 316LN even though oxygen contents are similar (a), and how the impact toughness can vary depending on manufacturing process parameters for the same batch (b). Relatively large differences in impact toughness can be observed for PM HIP 316LN between different batches and process parameters which indicate

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that the total oxygen content is not the only parameter to indicate this property in PM HIP materials. Note that some samples reach close to 400 J which is similar to the previously mentioned PM HIP 316L with greatly reduced oxygen content (22 ppm) [16]. This highlights that good impact toughness can be achieved with 100 ppm oxygen content, i.e. without having to greatly reduce oxygen content.

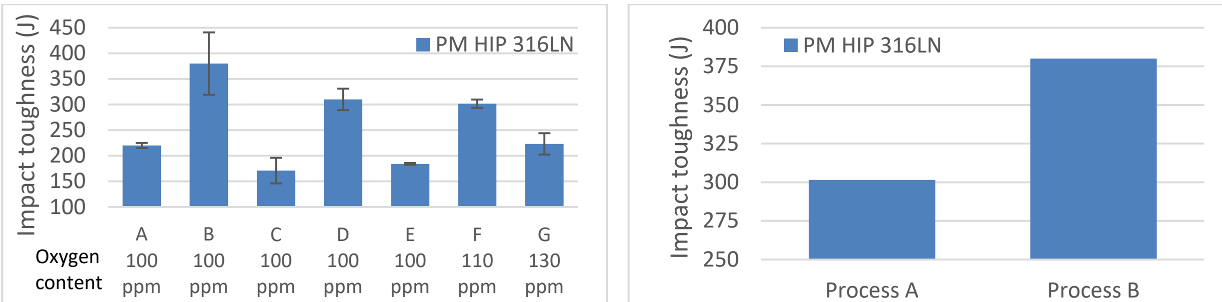

Fig. 9. *Samples with similar oxygen content (a), effect of process parameters on same batch.*

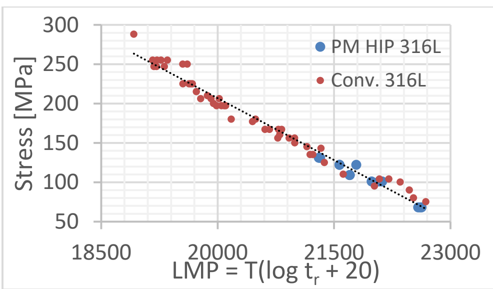

Fig. 10. *Creep properties.*

In Fig. 10. creep results for a PM 316L sample of the same batch as P1 [2, 3] HIPed at 1125ºC/100 MPa for 2 hours is presented as stress versus Larsson-Miller parameter (LMP) and compared to data for conventionally manufactured 316L. T is test temperature in Kelvin and t_r is hours to rupture. As can be observed the results are similar for PM HIP and conventional 316L. Test specimens were connected in series in test cells which were loaded prior to heating. The samples were at different instances removed from the furnace, unloaded and cooled down for measurements. No continuous measurements of load and elongation was available in the test setup.

Summary

PM HIP 316L/LN exhibits a homogeneous and isotropic microstructure with high cleanliness. Non-metallic inclusions found in the microstructure are small and relatively evenly distributed. Oxygen containing inclusions can seemingly affect the mechanical properties, both positively as in the case of yield strength, and adversely in some cases for impact toughness. The total oxygen content in PM HIP 316L/LN can on a broader scale indicate impact toughness levels, but results of this study shows that it is not the only parameter for this. Results presented in this study shows that excellent properties can be achieved for PM HIP 316L/LN at moderate oxygen levels if processed correctly.

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