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Effect of Processing Parameters on Intermetallic Phase Content and Impact Toughness for Super Duplex Alloy PM HIP Sandvik SAF 2507™

Martin Östlund[1,a *], Linn Larsson[1,b] and Tomas Berglund[2,c]

[1]Sandvik Materials Technology, Storgatan 2, 811 34 Sandviken, Sweden

[2]Sandvik Powder Solutions, Kontorsvägen 1, 735 31 Surahammar, Sweden

[a]martin.ostlund@sandvik.com, [b]linn.larsson@sandvik.com, [c]tomas.berglund@sandvik.com

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Abstract. PM HIP is a widely applied manufacturing technology to produce thick walled and complex shaped duplex and super duplex stainless steel (DSS and SDSS) components for the petrochemical as well as the oil and gas industry. The PM HIP process offers the advantage of a fine-grained microstructure which generates an increased resistance to HISC (Hydrogen Induced Stress Cracking) as well as higher yield strength. A limiting factor when producing thick walled components of DSS and SDSS alloys is the precipitation of brittle intermetallic phases which results in decreased corrosion resistance and impact toughness if high enough fractions are precipitated. The precipitation of intermetallic phases is a diffusion controlled process that may take place during quenching following solution annealing if the cooling rate is too slow. The cooling rate during quenching is mainly depending on the section thickness of the component, where large sections are subjected to slower cooling and thus increased intermetallic phase precipitation. In this article, it is shown that a coarser PM HIP microstructure results in lower contents of intermetallic phases after water quenching. However, despite of the lower intermetallic phase content the impact toughness is not improved and this is explained by the fracture mechanisms as shown by instrumented impact testing and fracture surface analysis.

Introduction

Sandvik SAF 2507™ is a super duplex stainless steel characterized by excellent resistance to stress corrosion cracking, pitting and crevice corrosion, general corrosion and high mechanical strength. Increasing water depths (increasing pressures) and increasing process temperatures in PM HIP applications for the oil and gas sector results in designs with increasingly large wall thickness. A limiting factor when it comes to increased wall thickness is the formation of brittle intermetallic phase during water quenching following heat treatment. The intermetallic phases nucleate and grow primarily in ferrite grain boundaries and ferrite/austenite phase boundaries in the approximate temperature interval 600 - 1000°C [1,2]. The thicker the manufactured component is, the longer times the center part of each section is subjected to the temperature interval in which intermetallic phase is formed during water quenching. Even smaller amounts of intermetallic phase content may affect the impact toughness adversely for DSS and SDSS components. This study was conducted to investigate if a coarser microstructure (i. e. reduced grain and phase boundary area) obtained by higher HIP temperature could result in lower amounts of intermetallic phase along with improved impact toughness of thick walled components of PM HIP SAF 2507.

Experimental

Two mild steel capsules with dimensions Ø133x250 mm and two with dimensions Ø236x250 mm were filled with SAF 2507 powder with composition per Table 1. The filled capsules were evacuated after which one of each capsule type were HIPed at 1150°C and 100 MPa with 3 hours holding time. The remaining two capsules were HIPed in another HIP cycle at 1250°C and 100 MPa with 3 hours holding time. The HIPed capsules were tested with regard to Argon content to verify that no capsule leakages occurred during HIP. Once it was verified that no detectable argon was present in the HIPed capsules they were heat treated. Capsule ID, HIP and heat treatment details can be seen in Table 2.

Table 1. *Chemical composition [wt%] of SAF 2507 powder batch.*

C	Si	Mn	P	S	Cr	Ni	Mo	Cu	N
0.015	0.41	0.80	0.018	0.002	25.0	6.85	3.82	0.09	0.28

Table 2. *Capsule, HIP and heat treatment details.*

Capsule ID	HIPed capsule size	HIP parameters	Heat treatment parameters
1191-1	~Ø120x220 mm	1150°C/3h/100 MPa	1070°C/4h/WQ
1192-1	~Ø210x220 mm	1150°C/3h/100 MPa	1070°C/5.75h/WQ
1191-2	~Ø120x220 mm	1250°C/3h/100 MPa	1070°C/4h/WQ
1192-2	~Ø210x220 mm	1250°C/3h/100 MPa	1070°C/5.75h/WQ

Three Charpy V-notch impact test bars were prepared from each of the HIPed and heat treated capsules at half height and surface, half radius and center position respectively. The manufactured test bars were tested by instrumented impact testing at -46°C per ASTM 2298. The CPT (Critical Pitting Corrosion Temperature) was measured per ASTM G150 on tested impact test bars from surface and center locations of capsules 1192-1 and 1192-2.

EBSD data collection was performed at 500x magnification on the non-deformed microstructures of ruptured impact test bars at surface, half radius and center position of the Ø210 mm capsules (1192-1 & 1192-2). The amount of austenite, ferrite and sigma phase was measured. The grain size was determined as area weighted average equivalent circle diameter (ECD) and as linear intercept grain size was from the EBSD images using 50 equidistant horizontal and vertical lines. Grain detection was performed disregarding sigma 3 twin boundaries in both cases. The EBSD data collection details can be seen in Table 3 and Fig. 1.

Table 3. *EBSD data collection details.*

Parameter	Setting
Camera resolution	461 x 345 pixels
Binning	4 x 4 (160x120 pixels)
Exposure time	13.8 ms
Gain	15
Band detection	12
Hough resolution	60
Step size	0.5 µm
Image size	0.23 x 0.17 mm = 0.039 mm^2

Fig. 1. *EBSD site.*

In addition to EBSD quantification of sigma phase and grain size measurements, the amount of intermetallic phase and austenite spacing was determined on the center samples of capsules 1192-1 and 1192-2 using image analysis. The samples were polished and etched in Murakami´s etchant after which image analysis at 500x magnification was conducted on 20 random fields of view.

Results

The results from the impact testing at -46°C showed similar impact toughness at centre, half radius and surface positions for the investigated capsule sizes regardless of HIP-temperatures. Although there were no large differences, slightly lower impact toughness is indicated for capsules HIPed at 1250°C compared to the capsules HIPed at 1150°C. Previous studies conclude that the reduction in impact toughness at centre position of the Ø120 mm capsules and half radius and centre position of the Ø210 mm is due to intermetallic phase content [1,3,4]. The results from the impact testing can be seen in Fig. 2 along with previous results for the same powder batch [3] where the average values of three samples are presented with the standard deviation as error bars.

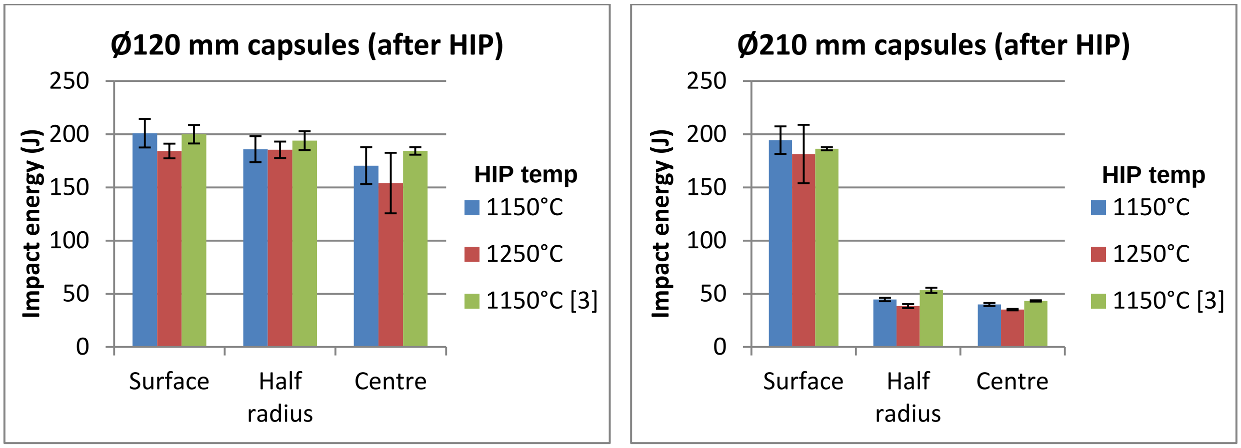

Fig. 2. *Impact toughness results at -46°C for Ø120 mm and Ø210 mm capsules HIPed at 1150°C and 1250°C.*

Some differences can be observed in the load vs. deflection curves of selected test specimens of each capsule obtained from the instrumented impact testing which is shown in Fig. 3. For surface samples in both Ø120 mm and Ø210 mm capsules it can be observed that HIP at 1150°C generates fully ductile fracture while HIP at 1250°C generates a ductile-brittle fracture. For the mentioned samples HIPed at 1250°C a brittle region can be observed (vertical drop) after crack initiation (i. e. after peak load) leading to lower crack propagation energy and lower impact toughness in total. For half radius and center samples in the Ø210 capsule, it can be observed that all samples exhibit a ductile-brittle fracture. Fracture surfaces of impact test bars tested at -46°C from surface position in 180x70x50 mm capsules HIPed at 1150°C and 1250°C followed by 1070°C/2h/WQ heat treatment is shown in Fig.4 and Fig. 5 respectively. These samples were produced from the same powder batch as the samples of this study [5]. In these figures, it can be observed that specimens HIPed at 1150°C exhibits a fully ductile dimple fracture while samples HIPed at 1250°C exhibits ductile dimple fracture with local areas of brittle fracture appearing to be fractures along grain and/or phase boundaries or alternatively cleavage fracture.

The amount of intermetallic phase and austenite spacing measured by image analysis at the center position of the Ø210 mm capsules can be seen in Table 4. All values are expressed as average values ± standard deviation. As can be seen it appears as if the capsule HIPed at 1250°C contains lower amounts of intermetallic phase, although the standard deviations are overlapping.

Table 4. *Intermetallic phase content (area percent) and austenite spacing.*

Capsule ID	HIP temperature	Intermetallic phase [%]	Austenite spacing [µm]
1192-1	1150°C	0.197 ± 0.096	8.7 ± 5.2
1192-2	1250°C	0.112 ± 0.076	13.2 ± 8.1

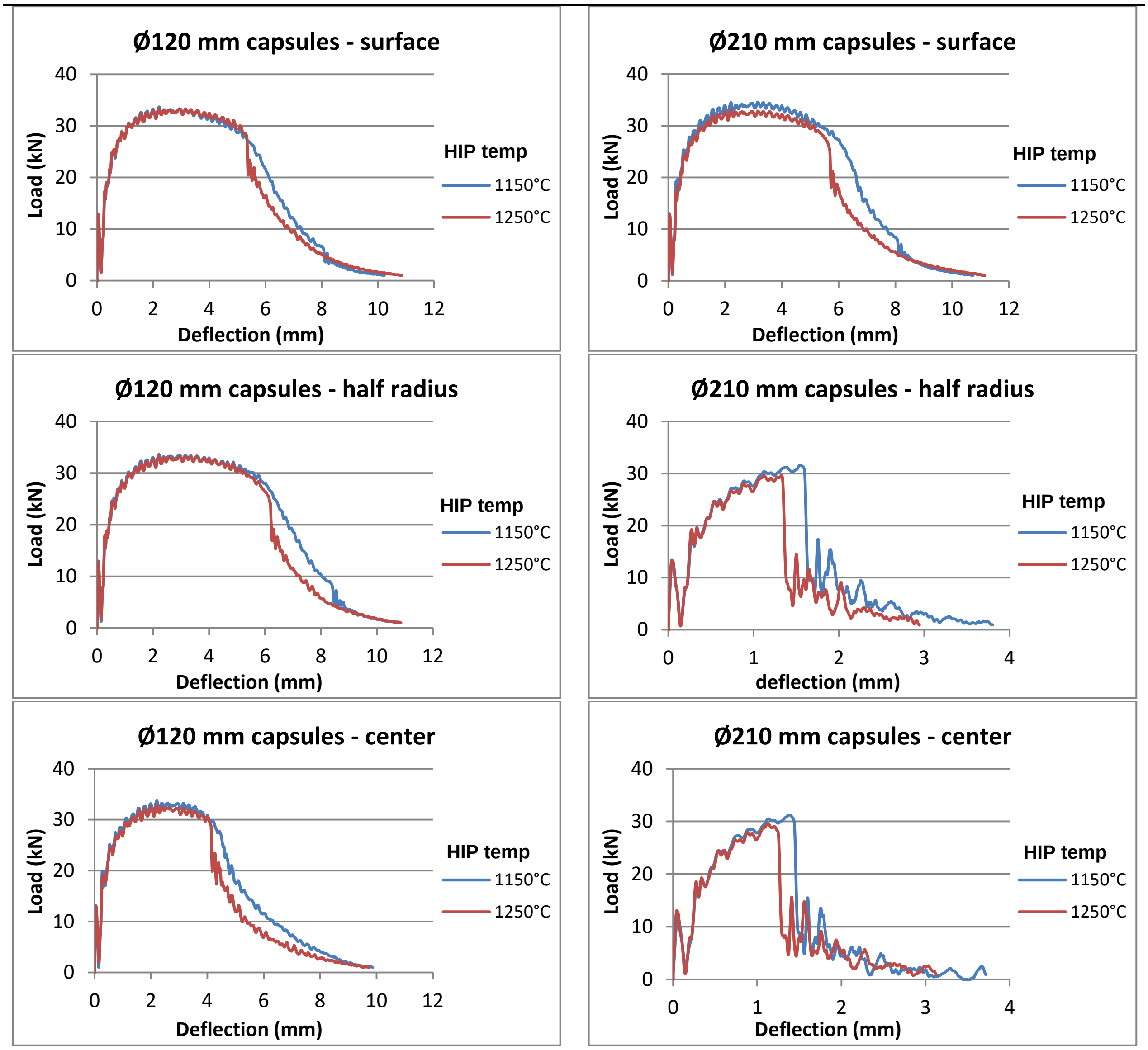

Figure 3. *Load vs. deflection curves from the instrumented impact testing at -46°C.*

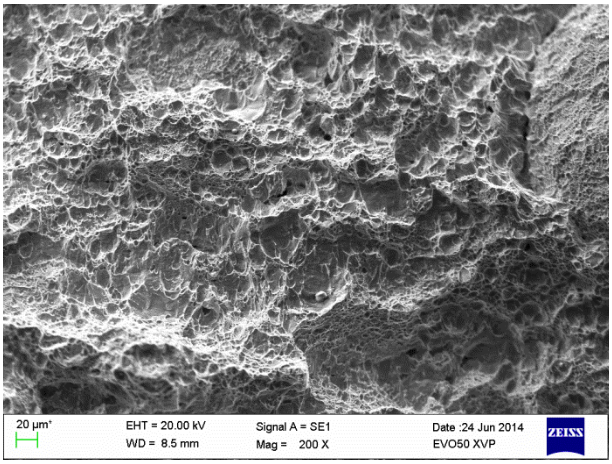

Fig.4. *Fracture surface of impact specimen HIPed at 1150°C, 200x magnification [5]*

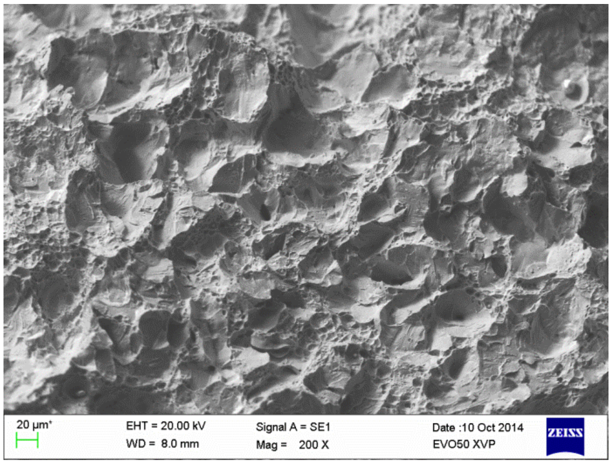

Fig. 5. *Fracture surface of impact specimen HIPed at 1250°C, 200x magnification [5]*

Grain size measurements from the EBSD data collection at center, half radius and surface location of the Ø210 mm capsules can be seen in Table 5. As can be noted the grain size of the capsule HIPed at 1250°C is approximately 60 – 65 % larger than for the capsule HIPed at 1150°C. The grain size is generally larger in the austenite (FCC) compared to the ferrite (BCC) regardless of HIP temperature. Another observation is that the grain size at the center seems to be slightly smaller (3 – 6 %) than the grain size at half radius and surface positions.

Table 5. *Grain size from the EBSD data collection presented as average values.*

Capsule ID	Phases	Area weighted ECD [µm]			Line intercept [µm]		
		Surface	Half radius	Center	Surface	Half radius	Center
1192-1	All	12.15	12.22	11.85	7.20	7.14	6.75
	FCC	12.54	12.59	12.33	7.82	7.81	7.33
	BCC	11.66	11.69	11.18	6.54	6.39	6.14
1192-2	All	20.08	19.72	19.18	11.70	11.89	11.10
	FCC	20.91	19.72	19.99	13.45	12.62	11.44
	BCC	18.97	19.72	18.04	9.96	11.13	9.87

Images from the EBSD data collection of mentioned samples can be seen in Fig. 6 where the austenite (FCC) phase is marked in blue, ferrite (BCC) in red and sigma phase in black color. From the EBSD data collection results it appears as if the material HIPed at 1250°C generally contains lower amounts of sigma phase. Since the measurements are only conducted on single fields of view there is however no statistical basis to support this. The average CPT from the ASTM G150 corrosion testing is detailed in Table 6. Similar values are obtained for all samples. The results are in line with previous results from both PM HIP SAF 2507 and conventionally produced SAF 2507 (80-90°C) [1,2].

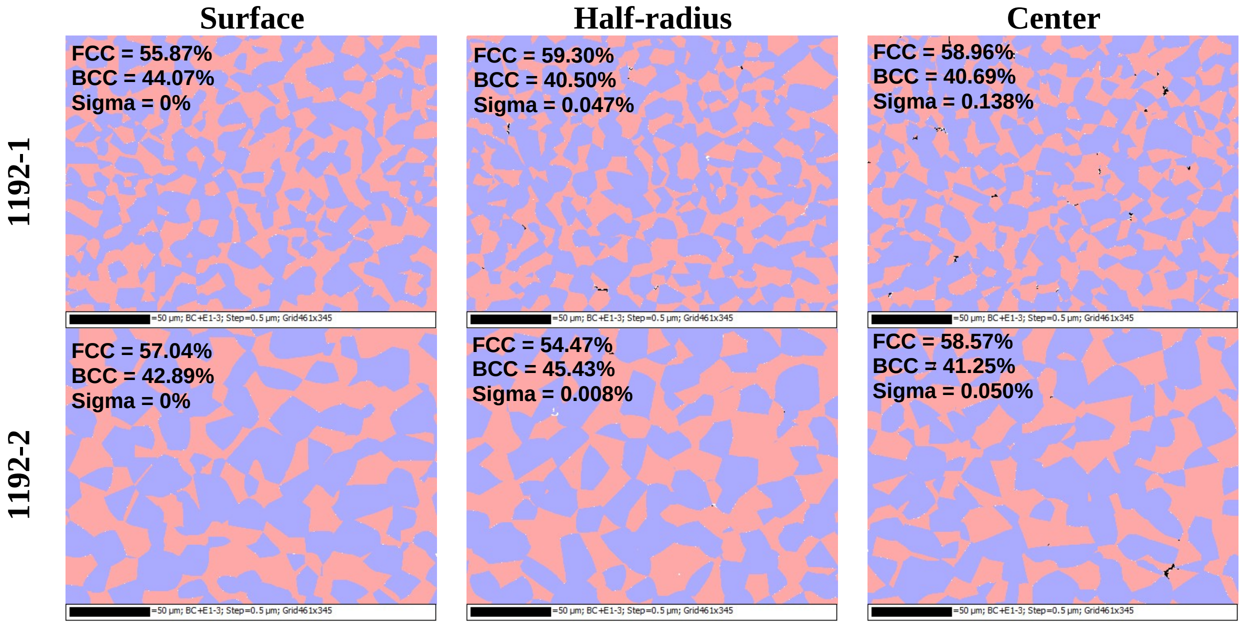

Fig. 6. *EBSD maps at center, half radius and surface positions in the Ø210 mm capsules (FCC = blue, BCC = red and Sigma phase = black), 500x magnification.*

Table 6. *Average CPT from ASTM G150 testing.*

Sample ID	Critical Pitting Corrosion Temperature [°C]	
	Capsule surface	Capsule center
1192-1	88.0	90.5
1192-2	88.4	89.2

Discussion

The results of this study indicate that a lower amount of intermetallic phase is formed in PM HIP SAF 2507 when produced with higher HIP temperature. The measured differences are however small and the standard deviations of the mean values are overlapping. It should be observed that lower amounts of sigma phase are measured with EBSD compared to intermetallic phase content measured by image analysis. A likely explanation to this could be that the EBSD analysis only measures sigma phase while the image analysis measures all intermetallic phases, i.e. also includes any eventual χ-phase. The intermetallic phase content can also be over-estimated due to etching effects while it can be underestimated with EBSD due to the inadequate resolution from the selected step size. There are some non-indexed points in the EBSD maps surrounding the sigma phase areas which suggest this. Beside these factors there is also the obvious difference in number of fields of view (1 vs 20), i.e. the EBSD measurements are more uncertain. A likely explanation for smaller amounts of intermetallic phase in the samples HIPed at 1250°C would be the coarser microstructure. A coarser microstructure leads to reduced grain and phase boundary area and ultimately result in fewer possible locations/smaller area for nucleation and growth of intermetallic phase.

Even though lower amounts of intermetallic phase are indicated for the capsules HIPed at 1250°C, the impact toughness is not improved compared to the capsules HIPed at 1150°C. By observing the load vs. deflection curves from samples free of intermetallic phase (surface samples of Ø120 and Ø210 mm capsules), it can be observed that 1150°C HIP results in a fully ductile fracture while 1250°C HIP results in ductile-brittle fracture. This also coincides with observations at fracture surfaces where 1150°C HIP samples exhibits a fully ductile dimple fracture while 1250°C HIP samples exhibits ductile dimple fracture with local areas of brittle fracture. The brittle fractures seem to have propagated along grain and/or phase boundaries or alternatively they are cleavage fractures. The partial brittle fracture is likely an explanation for the lower impact toughness of samples HIPed at 1250°C, regardless of intermetallics, and seems to be correlated to larger grain size. The exact mechanism causing the brittle fracture is not fully understood and needs to be investigated further. The half radius and center samples of the Ø210 mm capsules is likely to contain the largest amounts of intermetallic phase due to slower cooling rates during water quenching. Even though the results of this study imply that the capsule HIPed at 1250°C contains lower amount of intermetallic phase content, the results in impact toughness are very similar. The explanation for the similarly low impact toughness levels is that the intermetallic phase content of these samples likely is too large in both cases.

Conclusion

Lower intermetallic phase content is indicated for PM HIP SAF 2507 HIPed at 1250°C compared to 1150°C. A probable explanation for this is the coarser microstructure, i.e. reduced grain and phase boundary area, which results in fewer locations for nucleation and growth of intermetallic phase. Samples HIPed at 1250°C free of intermetallic phase exhibits a ductile-brittle fracture during impact testing, which manifests itself as partial brittle fracture. This results in lower crack propagation energy and consequently lower impact energy compared to samples HIPed at 1150°C which exhibit fully ductile fracture with corresponding ductile dimple type fracture surface. Even though higher HIP temperatures might reduce the susceptibility towards formation of intermetallic phases, the impact toughness is not necessarily improved. The coarse microstructure itself seem to generate lower impact toughness regardless of intermetallic phase content.

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