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Toughness of Duplex Steel Produced by PM-HIP

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Abstract. The most important influencing factors for the toughness of duplex steels are being discussed exemplary at grade AISI 318LN. Focus is given to two major aspects: the embrittlement by σ-phase and the embrittlement caused by residual argon pores. While the formation of σ-phase depends on the cooling rate in the HIP vessel, argon porosity can either be caused by insufficient evaporation prior to HIP or small leakages in the capsule. Toughness is discussed in terms of Charpy tests, taking into account the notch radius as additional parameter. The macroscopic results are reflected by investigations of the microstructure. Toughness of PM-HIP steel is compared to appropriate conventionally produced grades.

Introduction

Components produced by PM-HIP from corrosion resistent steels with a ferritic-austenitic duplex microstructure are widely used in offshore-applications, in the food industry, in ship building and in the chemical industry [1]. In most of these applications high toughness – particular at low temperatures – is a mandatory requirement. In contrast to austenitic steels, toughness of duplex steel shows a temperature dependent transistion from ductile to brittle behaviour. This is caused by the transition from ductile dimple fracture to brittle transcrystalline cleavage at low temperatures within the ferrite grains. While PM-HIP duplex steels mostly have superior strength and corrosion resistance compared to conventionally produced grades, the toughness issue often leads to discussions.

Factors influencing toughness can be grouped into factors describing the loading and effects of the material's microstructure. While the former contain loading conditions which determine the thermal activation of deformation mechanism, like testing temperature and deformation rate as well as stress triaxiality, the latter factors comprise the crystal lattice, the grain size or non metallic inclusions. In case of duplex steels intermetallic phases strongly determine the toughness of the material. Particular the σ-phase which forms in the temperature range between 940°C and 750°C and the α'-phase which forms at temperatures about 450°C leads to embrittlement of duplex steel even at very small volume fractions [2, 3]. Particular in case of PM-HIP materials, toughness is influenced by two additional effects: the content of oxygen and the content of argon. Stable oxides at the surface of powder particles may not dissolve during HIP and remain as so called prior particle boundaries (PPB's). Bengtsson e. al. [4] revealed by a comparison of two duplex steel powders with different oxygen contents that 1470 ppm of oxygen lead to clearly visible PPB's while no oxides were detectable in hot isostatically pressed powder with 110 ppm. Hämöläinen [5] showed that the oxygen content drastically reduces the Charpy toughness of AISI 318LN duplex steel (see fig. 1).

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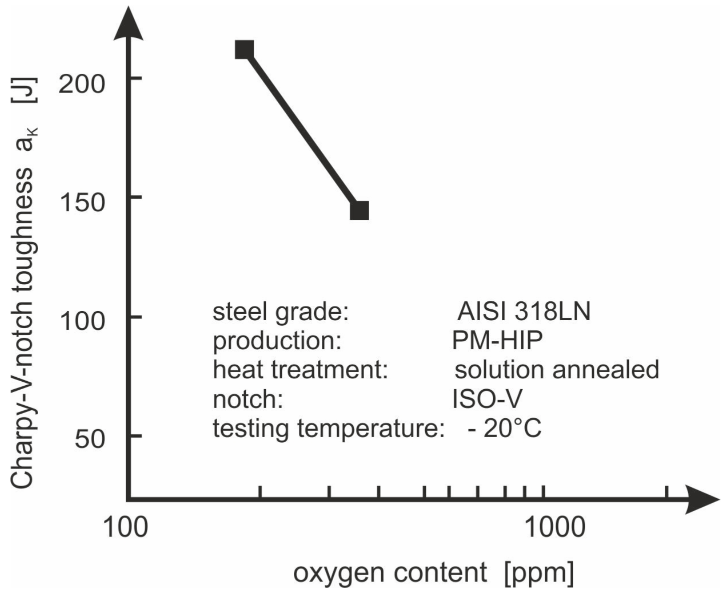

Figure 1: *Influence of oxygen content on Charpy-V-notch toughness, according to [5]*

The second HIP specific factor is Argon. The origin for Ar pores in PM-HIP material can either be contamination of Ar atomized powder or tiny leakages in the capsule. It must be mentioned that using modern production routes for PM-HIP parts, nowadays the argon content usually is below the detection limit. Nevertheless, as small amounts of Ar drastically reduce the toughness, knowledge on the mechanisms that lead to embrittlement by Ar is necessary.

In this study the effects of σ-phase, Ar-content and stress triaxiality has been examined. Moreover, the toughness of duplex steel AISI 318LN is compared to competing production methods like sand casting and open die forging.

Experimental

Toughness tests have been performed with duplex steel AISI 318LN (PM2205, ASTM F51, DIN 1.4462, X2CrNiMoN22-5-3) produced by PM-HIP, continuous casting+forging and sand casting. Tab. 1 gives the chemical composition of all three grades tested. The powder for the PM-HIP grade was gas atomized under nitrogen. The oxygen content of the powder was 85 ppm, its apparent density 4.69 g/cm^3 and its tap density 5.30 g/cm^3. The particle size distribution is given in tab. 2. Two types of capsules have been produced: D110mmxH235mm and D200xH235mm. Capsule material was AISI 304 stainless steel. The big capsules have been consolidated by HIP using a cycle with a holding temperature of 1140°C, a holding time of 200 min and a pressure of 1010 bar. Fig. 2 shows this HIP cycle with the temperature range for σ-phase included.

In order to produce samples with defined Ar-contents 5 “small” capsules have been filled with powder (relative filling density: 66-69%). After leak testing with He, these capsules have been 5 times evacuated down to 10^{-3} mbar and flushed with Ar (1.5 bar). The desired Ar-content was adjusted by the Ar pressure of the final flushing step. These capsules have been consolidated at 1140°C for a holding time of 240 min and a cooling rate of 10 K/min. The pressure was 1000 bar. The final Ar-content was characterized using a gas analyzing device type Extra-Werf with a minimum detection limit of 0.05 ppm. This device is based on gas chromatography.

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Table 1: *Chemical composition of the steel grades investigated*

production route	Chemical composition [weight- %]						
	C	Si	Mn	Cr	Mo	Ni	N
PM-HIP (powder)	0.015	0.68	1.02	22.20	3.09	5.19	0.170
continuous cast + hot forged	0.020	0.50	1.71	22.46	3.36	5.40	0.174
sand cast	0.029	0.72	1.35	23.07	2.78	6.5	0.181
EN 10088	≤ 0.03	≤ 1.0	≤ 2.0	20.0 – 23.0	2.5 - 3.5	4.5 – 6.5	0.10 – 0.22

Table 2: *Particle size distribution of the gas atomized steel powder*

particle size [µm]	500	250	125	106	63	45
fraction < [%]	100	84	47	37	18	10

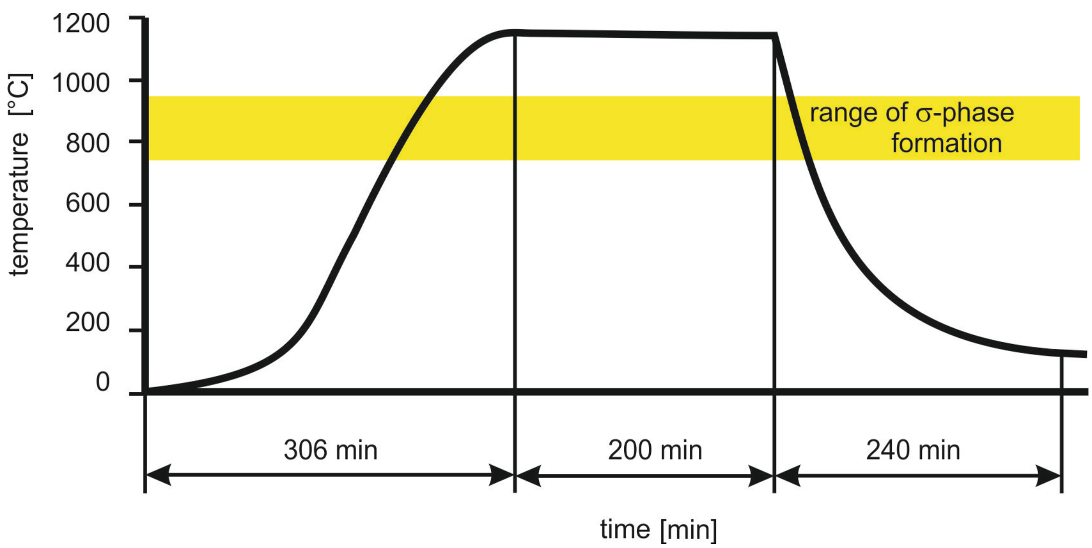

Figure 2: *HIP cycle for the production of samples in an industrial scale plant*

In order to characterize the microstructure light optical sections have been prepared by grinding with SiC paper and polishing with diamond slurry. The microstructure was revealed using Groesbeck-etching. Using this preparation prior to light optical microscopy the austenite appears light brown, the ferrite dark brown and σ-phase blue. The phase constitution was determined using EDS analysis in a SEM Typ Zeiss, Leo 1450VP. SEM was also used to investigate the fracture surfaces. The density of the consolidated samples was measured by He-pyknometry.

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Charpy notch toughness was measured using a pendulum hammer with maximum energy of 300 J. Three samples were tested per temperature, 296 samples were tested in total. The testing temperature was varied between -185°C and +400°C. For this purpose, the specimens were cooled in different liquids or heated in a furnace prior to testing. Liquid nitrogen has been used as cooling liquid for -185°C and a mixture of ethanol and dry ice for -40°C and -75°C, respectively. The temperature in the furnace or the cooling vessel was measured with a thermocouple directly attached to the specimen. After reaching the desired temperature the specimen was placed to the anvil of the hammer and immediately broken within a maximum time period of 3 seconds. Fig. 3 shows the geometry of the Charpy specimens. In order to study the effect of the stress triaxiality the notch radius was varied from 0.25 mm via 1.0 mm to 2.0 mm.

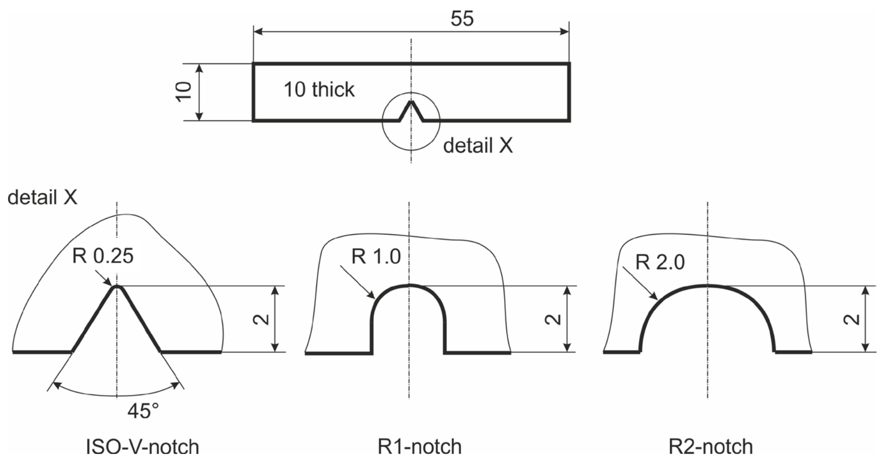

Figure 3: *Geometry of Charpy-notch-specimens*

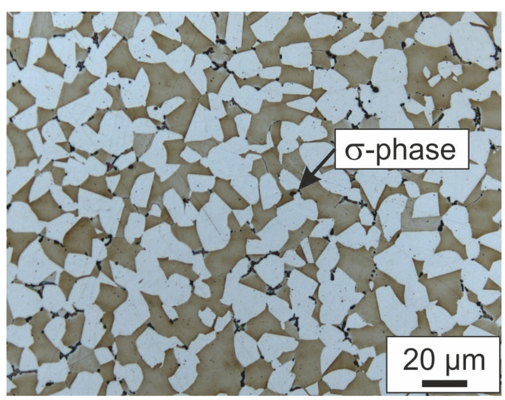

Figure 4: *Microstructure of the PM-HIP grade after HIP*

Results

The microstructure of PM-HIP 318LN steel in the as HIP condition is shown in Fig. 4. At the boundaries between ferrite and austenite grains the precipitation of σ-phase can be seen as well as at boundaries between ferrite and ferrite grains. In order to remove the σ-phase after HIP most samples were solution annealed at 1060°C for 3 h and subsequently quenched in water. By this

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heat treatment the σ-phase could be totally removed. The fractions of all phases were determined by image analysis using a magnification of 500:1 and 1000:1. A number of 3 micrographs corresponding to a total area of 44 mm^2 for the as HIP'ed state and 131 mm^2 for the annealed state were analyzed. The resulting volume fractions are given in tab. 3 for the as HIP state and for the solution annealed steel.

Table 3: *Phase fractions in PM-HIP duplex steel, depending on heat treatment*

state	phase fraction [vol.- %]		
	austenite	ferrite	σ-phase
as HIP	50.9 ± 1.3	46.8 ± 2.2	1.3 ± 0.2
HIP + solution annealed	50.5 ± 1.4	49.4 ± 1.4	0

In [6] the composition of each phase has been determined using EDS analysis. The ferrite dissolves about 3.7% more chromium and 2.8% less nickel compared to the austenite. 3% Ni and 8% Mo are dissolved in the σ-phase.

Fig. 5 shows the microstructure of the PM-HIP duplex steel in comparison to material produced by alternative production technologies. Obviously, the forged grade is characterized by a remarkable anisotropy. The individual grains are elongated in the direction of hot forming as seen in the longitudinal section. The transverse section reveals a dispersion of austenite in the ferrite. The microstructure produced by sand casting is isotropic but is determined by a coarse dendritic structure of the austenite.

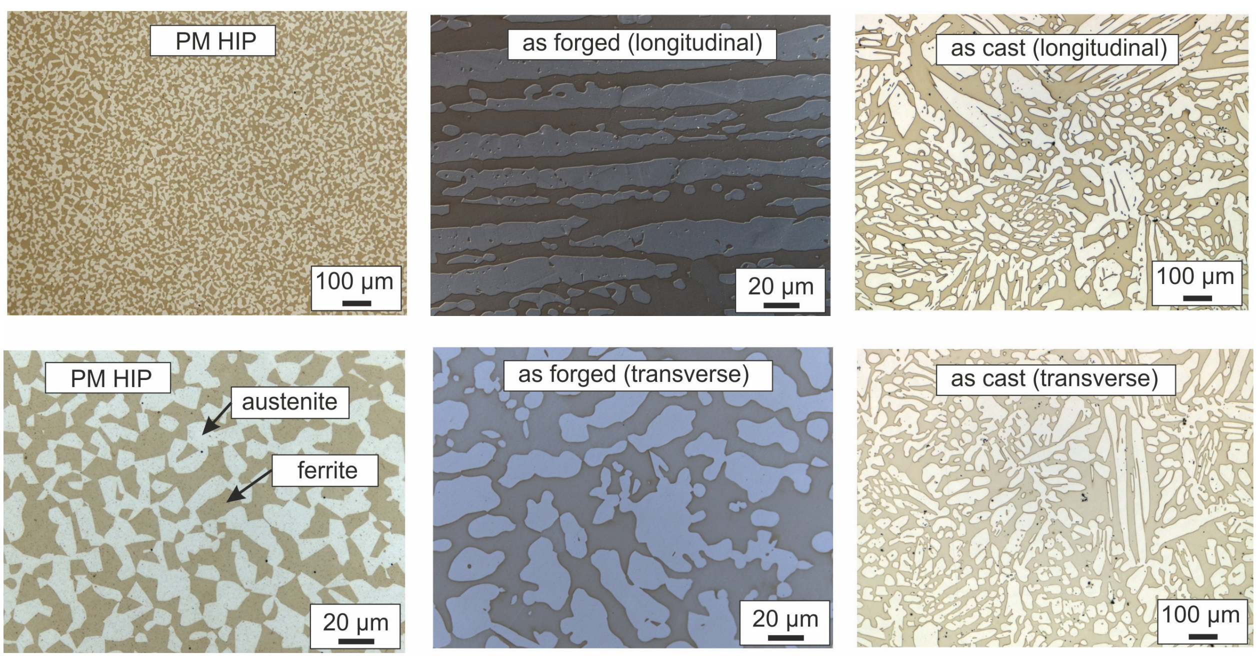

Figure 5: *Microstructure of steel AISI 318LN after solution annealing;*
left: PM-HIP, middle: as forged; right: as cast

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Charpy toughness a_k in this study is plotted against the testing temperature. This allows to discuss the influence of the different effects on toughness in terms of the height of the upper shelf and the shift of the brittle to ductile transition temperature (BDTT). The measured energy to fracture in all diagrams showing toughness results is related to the net cross section of the samples. Fig. 6 shows that the presence of only 1.3 vol.-% of σ-phase in the as HIP state reduces the toughness drastically: Toughness in the upper shelf with 90 J/cm^2 is about only one third of the appropriate level of 312 J/cm^2 in the precipitation free state. This illustrates that components with thick cross sections made of PM-HIP duplex steel need to be post heat treated prior to service. The local stress triaxiality in the notch root has been varied in fig. 6 by modifying the notch radius. As can be expected an increase of the notch radius leads to a distinctive increase of the upper shelf toughness and a slight shift of BDTT towards lower temperatures. The relevant testing temperature for multiple applications in the offshore industry is -44°C. Although the standard ASTM A988 [7], which gives specifications for mechanical properties for stainless and duplex steel produced by PM-HIP, does not explicitly specifies a minimum toughness value, the toughness plotted in fig. 6 at -44°C should fulfil the requirements of most applications.

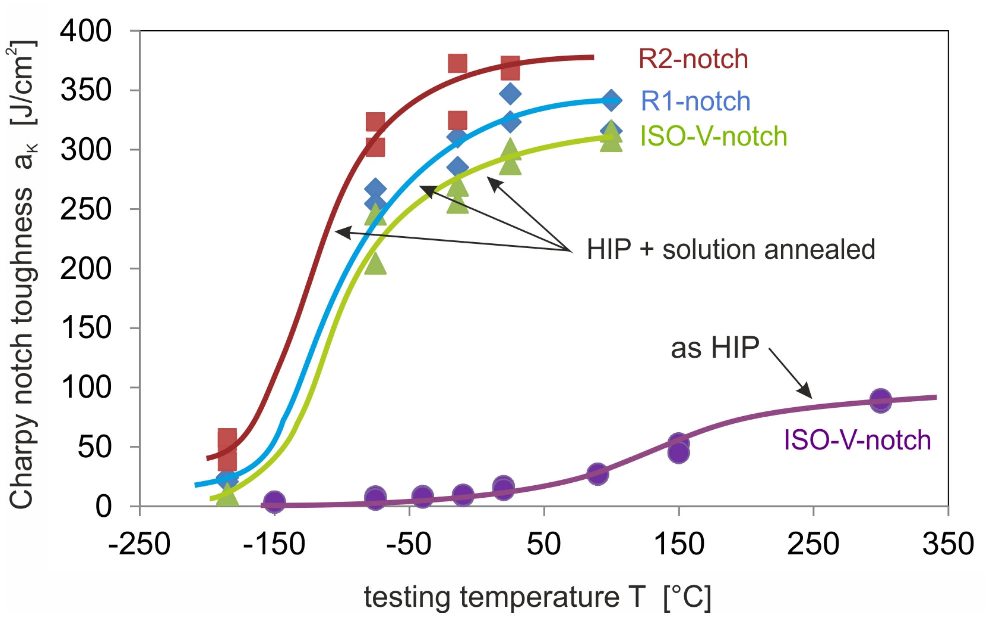

Figure 6: *Charpy toughness of PM-HIP duplex steel – influence of heat treatment and notch geometry*

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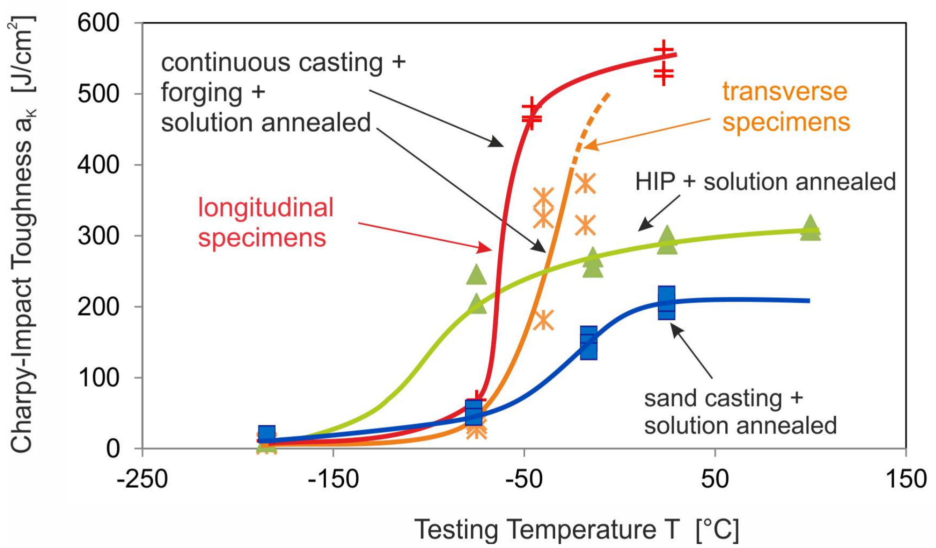

Figure 7: *Charpy toughness of AISI 318LN duplex steel: Comparison of production methods, notch geometry for all specimens: ISO-V-notch*

Fig. 7 shows the influence of the production method on the toughness. The lowest toughness was measured with samples produced by sand casting. Although a level of approx. 200 J/cm^2 in the upper shelf represents still a tough material, this value is remarkably lower compared to the PM-HIP grade. Comparing those two routes, the BDTT is lower in the as HIP material and in contrast to the cast grade gives sufficient toughness even at -44°C. The highest toughness is achieved with the forged material. The disadvantage of forged steel is that toughness reduces very rapidly as soon the testing temperature falls below BDTT. This leads to the effect that the PM-HIP steel seems to be superior in applications which run at very low temperatures. As has been investigated in [6] the microscopic appearance of the fracture surface corresponds to these toughness curves: While the PM-HIP grade even in the transition area shows high amounts of ductile dimple failure, cleavage of the ferrite dominates the fracture surface of the forged grade in this temperature range. At very low temperatures only cleavage of ferrite grains is visible at all production routes: Consistently the toughness in the lower shelf does not depend on the production method.

The production of samples with artificial Ar contents lead to voids, filled with Argon, which appear circular in metallographic sections. Fig. 8 shows Ar pores in samples containing 1.0 ppm and 7.1 ppm Ar, respectively. At higher resolution (fig. 9) it can be seen, that at some regions the Ar voids seem to be arranged like a pearl necklace along the former particle boundaries. During HIP crystallographic grains grew across the former particle boundaries proving that the consolidation process performed perfectly.

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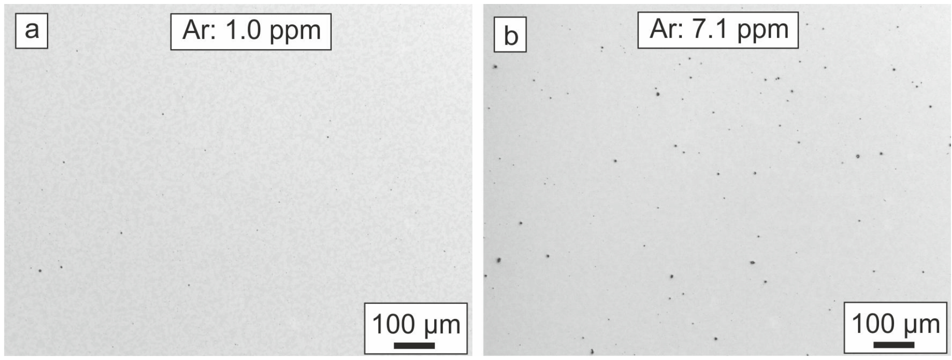

Figure 8: *Light optical section of HIP samples with different Ar contents*

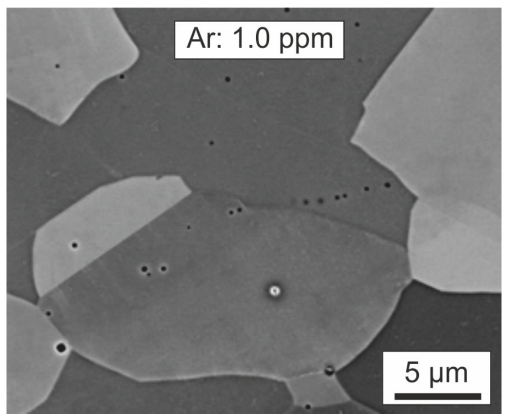

Figure 9: *SEM section of a HIP sample with 1.0 ppm of Ar*

Ar voids drastically reduce the toughness of PM-HIP duplex steel, as figures 10 and 11 point out for samples with two different notch geometries. The green lines represent the condition free of Argon. “< 0.05 ppm” means that the samples contain less Ar compared to the detection limit of the analyzer used. It can be expected that the Ar level is much lower. Already 0.52 ppm reduces the Charpy toughness in the upper shelf and leads to a shift of BDTT towards higher temperatures. 93 ppm of Ar lead to brittle fracture even at higher testing temperature.

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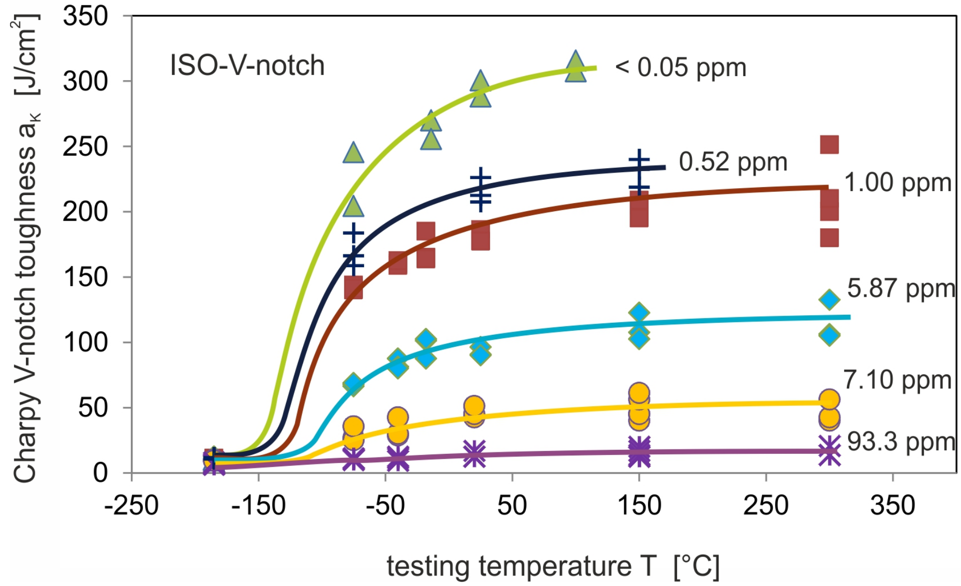

Figure 10: *Influence of Argon content on the Charpy notch toughness, ISO-V-notch*

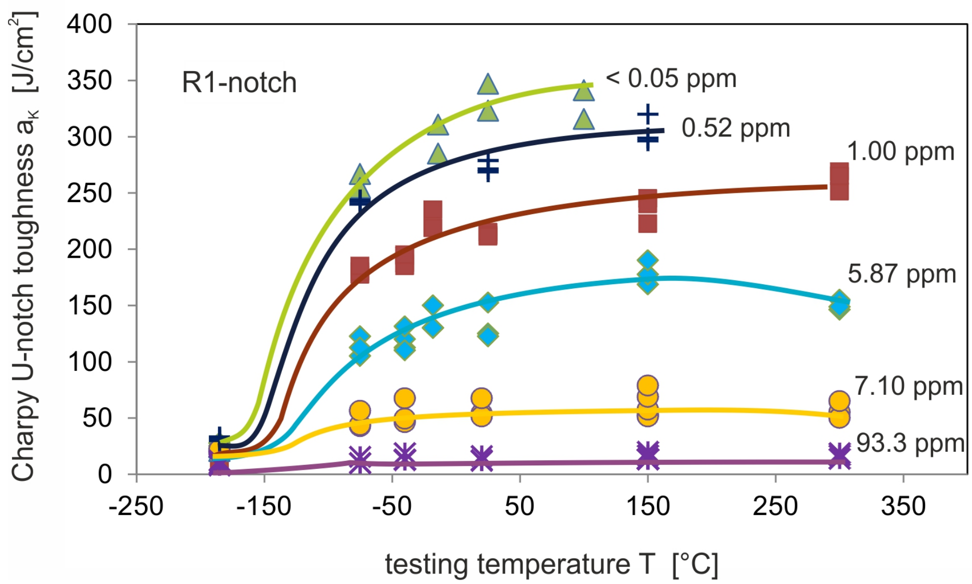

Figure 11: *Influence of Argon content on the Charpy notch toughness, R1-notch*

The fracture mechanisms can be studied by looking onto the fracture surfaces of broken Charpy-samples. Fig. 12 shows the fracture surface of a sample with 1.0 ppm Ar, tested at a temperature of 150°C. The high fracture energy indicates a ductile fracture mechanism. This is proven by dimples at the fracture surface, occurring in both phases ferrite and austenite. Individual Ar voids coalesce and form crack like carves (fig. 12 a). Individual Ar pores in the fracture surface no longer appear as spheres, but form a crack like pattern as seen in fig. 12 c. It

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can be assumed that trapped Ar under high pressure supports this transformation into microcracks during mechanical loading of the material. The same pattern is visible in the sample with 5.8 ppm Ar, tested at 150°C. Although the fracture energy is less compared to the specimen with 1.0 ppm Ar, the microscopic fracture appearance shows still ductile dimples in both phases. Fig. 13 shows microcrack growth including crack tip blunting and local crack opening by plastic deformation. Again it can be assumed that Ar was trapped under high pressure in the pore.

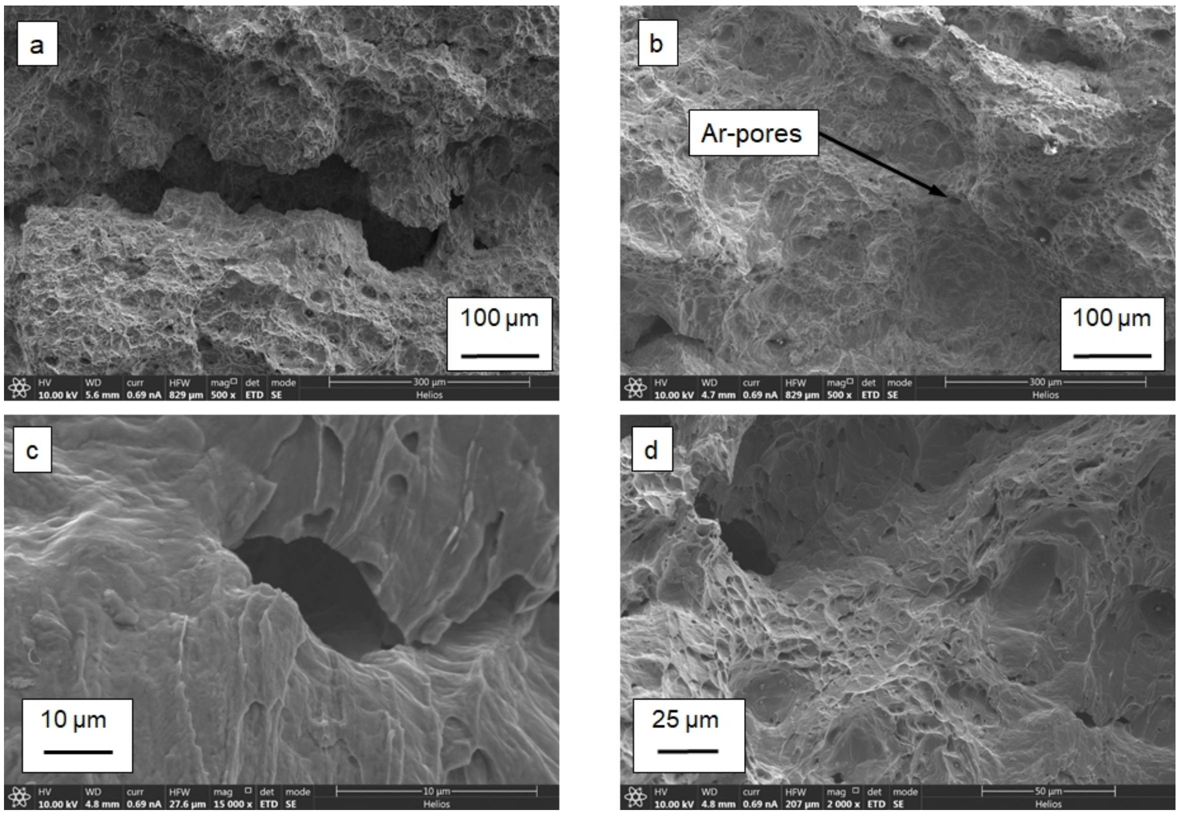

Figure 12: *Fracture Surface (SEM) of a PM-HIP sample with 1.0 ppm Ar, testing temperature T = 150°C,* a_k*=201 J/cm*^{2}

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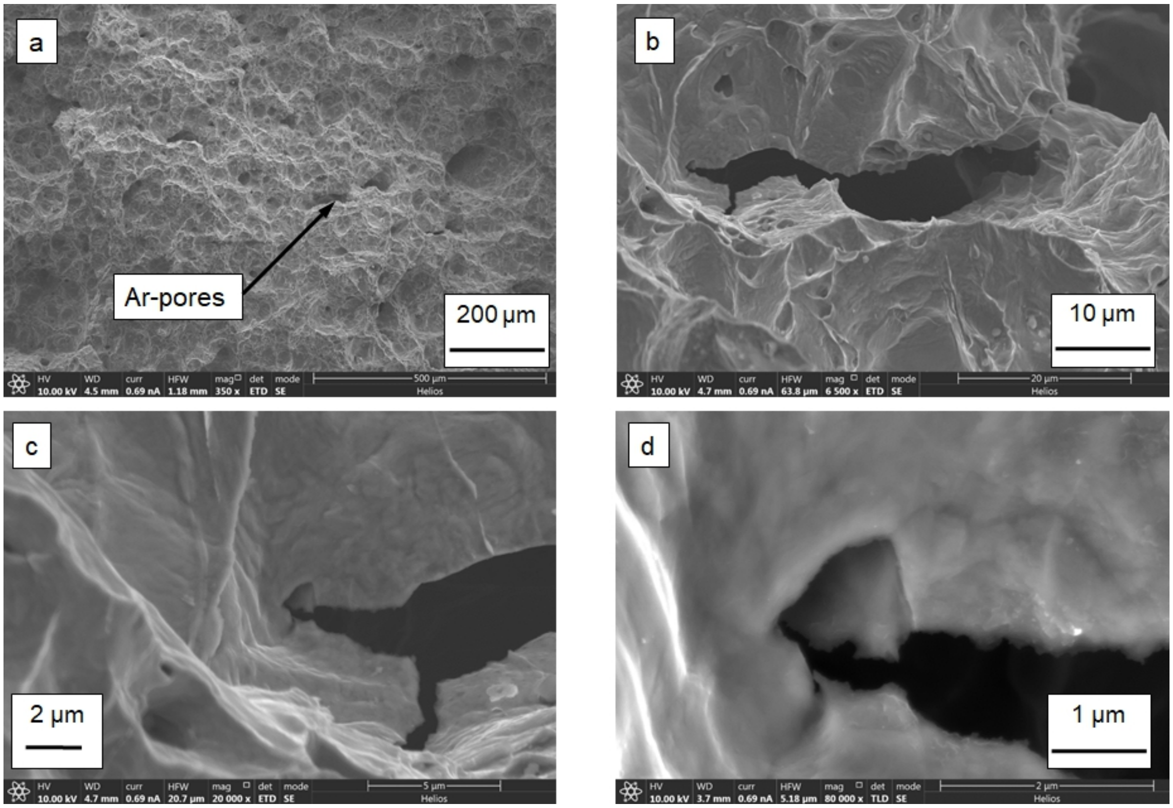

Figure 13: *Fracture Surface (SEM) of a PM-HIP sample with 5.8 ppm Ar, testing temperature T = 150°C, a_k=91 J/cm^2*

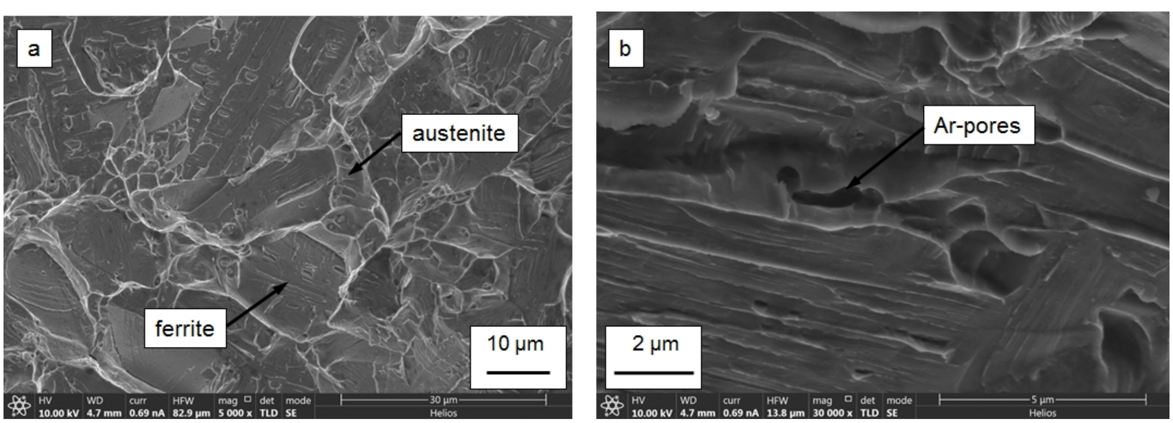

Figure 14: *Fracture Surface (SEM) of a PM-HIP sample with 1.0 ppm Ar testing temperature T = -185°C, a_k=11 J/cm^2*

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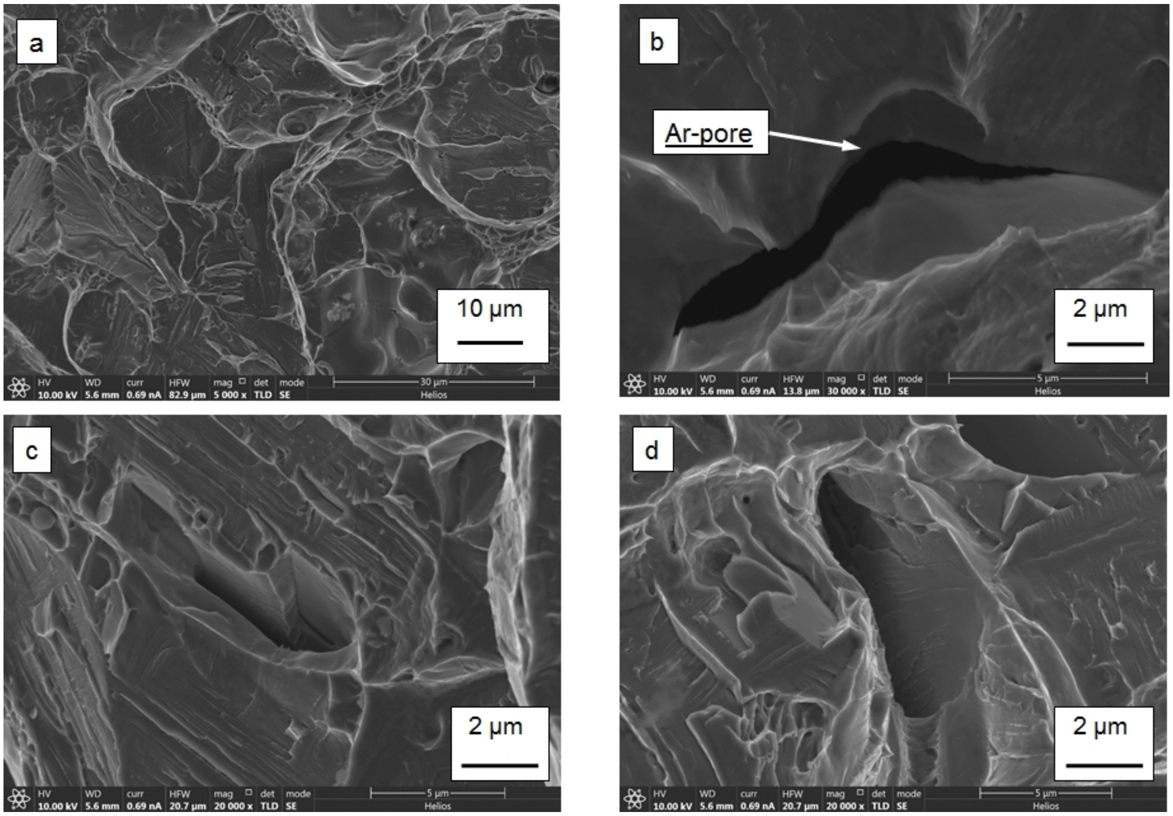

Figure 15: *Fracture Surface (SEM) of a PM-HIP sample with 5.8 ppm Ar, testing temperature T = -185°C,* a_k*=8 J/cm*^{2}

At low testing temperature the ferrite fails microscopically brittle by cleavage along crystallographic planes. In case of a sample with 1.0 ppm Ar, tested at -185°C, the biggest part of the fracture surface is covered by those cleavage planes. Some bridging areas through the austenite show local ductile fracture and shallow dimples (see fig. 14). Some Ar voids are visible at the fracture surface (fig. 14b). But in contrast to fracture under conditions leading to upper shelf fracture work, this Ar pore did maintain their shape during fracture and did not behave like a microcrack. It did not develop a sharp crack tip or typical signs of micro crack propagation. Finally, fig. 15 shows the fracture surface of a sample containing 5.8 ppm Ar and being tested at a temperature of -185°C. Again, only small areas of dimples are visible, indicating crack propagation through the austenite. Small lips, typical for cleavage fracture are seen in ferrite grains (see fig. 15 d). One Ar pore shows a crack like appearance (fig. 15 b), but does not reveal any signs of micro crack propagation. It can be concluded that trapped Ar does only slightly influence fracture in the low temperature regime below BDTT. Consistently the Charpy toughness in the lower shelf is only slightly depending on the Ar content.

Summary

The effects of manufacuturing method, heat treatment, notch geometry and Ar-content on the Charpy notch-toughness of AISI 318 duplex steel have been investigated. Independent of the testing temperature the toughness of PM-HIP steel is superior to cast material. PM-HIP material shows higher toughness compared to forged steel at temperatures lower than -50°C. Even a very small amount of σ-phase drastically reduces the toughness. A small notch radius leads to an increase of the BDTT and a decrease of the toughness level in the upper shelf. Toughness is very sensitive to the Ar content. Above BDTT Ar voids behave like microcracks which propagate during loading in the Charpy hammer. By this, small amounts of Argon decrease the fracture energy in the upper shelf of the toughness-temperature diagram. 93 ppm of Ar lead to brittle fracture even over the whole temperature range.

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