Microsegregation and High Temperature Properties of High Manganese Steels

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Abstract

High-manganese steels containing 10 to 30% Mn exhibit high strength and exceptional plasticity due to TWIP effect (TWinning Induced Plasticity) or via multiple martensitic transformations TRIP effect (TRansformation Induced Plasticity). High Mn steel also has low density because of Al addition and features high specific energy absorption. Therefore, it is a very promising material for the automotive industry. But it is hard to cast high Mn steel by the continuous casting process, and even by Horizontal Single-Belt Casting, as the slab is susceptible to cracking. This is because high Mn steel has poor hot ductility.

In order to design alloys with good ductility, a new shear test equipment was developed in the context of this work. It can test high temperature mechanical properties in as cast state solidified directly from the melt. In this way, the high Mn steels with good hot ductility can be selected and subsequently produced in the steel industry.

The reasons why some high Mn steels are prone to cracking are complex. High Mn steel with high carbon content has severe microsegregation, especially regarding Si. Furthermore with carbon content increasing, carbide formation increases and liquidus temperature decreases.

Engineering stress and true stress of high Mn steel at 1000 ℃ were investigated. It has to be point out that high elongation does not necessarily mean good hot ductility. In the experiments, samples that have cracks can also exhibit high elongation at high temperature.

After cooling down to room temperature and subsequently annealing for 1 hour at 1020℃ in the muffle furnace, hot ductility of high Mn steel becomes much better. For the Horizontal Single-Belt Casting process, casting of high Mn high carbon steels can be processed, because the strip has no bending during the casting. After the

casting, the strip should not be inline-rolled directly, but should first be reheated, and then rolled. For the traditional continuous casting process, it is not recommended to cast high Mn high C full austenite steels. If these are needed, the possible process is to cast ferritic+austenitic steel firstly, and subsequently anneal it in order to remove the ferrite phase.

Table of Contents

1 **Introduction**

High Mn steels offer excellent mechanical properties, compared to other steel grades. Despite of this, their industrial use is very limited. This is due to the fact that they are difficult to cast with large scale industrial processes, especially because of their poor hot ductility. Lots of research has been carried out about high Mn steels. But there are limited publications on high temperature properties of high Mn steel, especially for the as cast state. The as cast properties are relevant for continuous casting, strip casting and in-line rolling.

The aims of this research are as follows:

a) Understanding the mechanisms leading to embrittlement of high Mn steels at high temperature;
b) Development of techniques that can test hot ductility in the as cast state;
c) Getting hint for improving alloy design.

In the following chapter, the state of the art for the background of this work is given including a review on high Mn steel, especially on its high temperature properties, is given. Experimental methods, in particular hot shear test, are introduced in chapter 3. In chapter 4, experimental results of high Mn steel containing 13%[1], 15%, 17%, and 21% Mn are presented. Five different aspects about high Mn steel are discussed in chapter 5. Conclusions and outlook are given in chapter 6, while an appendix and references are presented in chapters 7 and 8 respectively.

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[1] All contents in this work are given in wt.-% unless otherwise noted.

2 Literature review

2.1 Automotive steel

In the automotive industry, greenhouse gas emission reduction and improvement of vehicle safety have been the main goals. As automakers are always trying to improve safety and reduce fuel consumption, they search for new materials to make a lightweight and strong vehicle [1] [2].

Fig. 2-1: Tensile properties of different steels [3].

Different types of steels have been used in the automobile industry. Their tensile properties are given in Fig. 2-1. A brief introduction of each steel type is summarized below.

a) Mild steel

Tamarelli said in [4] : conventional mild steel has a ferritic microstructure with low carbon content and minimal alloying elements in order to make it formable. Widely produced and used, mild steel is not expensive and often served as a baseline for comparison with other materials. Its strength is relatively low. It has been used for

many applications in vehicles, including the body structure, closures, and other ancillary parts.

b) IF steel

Singh said in [5] : IF (Interstitial Free) steel is free of interstitial atoms, mainly carbon and nitrogen in ferrite matrix and has excellent formability. Due to lack of any interstitial atoms in the solid solution, it is essentially non-aging. Main automotive applications of the IF steel are the rear floor pan, the spare wheel well, front and rear door inners, and closures.

c) BH steel

Senuma said in [6] : BH (Bake Hardenable) steel sheet is ideal for outer panels of cars because it simultaneously provides high formability, high surface deflection resistance. The features of BH steel sheet are that it is as highly formable in press forming as mild steel and is subsequently hardened by baking treatment process. Its yield strength can be increased by a minimum value of 30 MPa by the baking treatment at 170 ℃ for 20 min. This increase in strength is due to fixing the dislocations by interstitial atoms such as carbon and nitrogen in solution.

d) HSLA steel

Illescas said in [7] : HSLA (High Strength Low Alloy) steels can provide a better combination of strength, toughness and weldability than conventional carbon steels. They have yield strengths greater than 275 MPa. The chemical composition of a specific HSLA steel may vary for different product thicknesses to meet the requirements of mechanical property. The HSLA steels in slab form have low carbon content (0.05 to −0.25% C) in order to produce good formability and weldability, and they have Mn content up to 2.0%. Small quantities of chromium, nickel, molybdenum, copper, nitrogen, vanadium, niobium, titanium, and zirconium are used in different combinations [8]. Many automotive ancillary parts, body structure, suspension, and wheels, are made of HSLA steel [4].

e) Rephos steels

Mazancova said in [9]: Rephos steel has higher phosphorus content, which can increase matrix strength properties because of its high atomic diameter. Compared with HSLA, Rephos steel has higher formability.

f) DP steel

Tasan said in [10]: DP (Dual Phase) steel has ferritic-martensitic microstructure, which provides a wide range of excellent mechanical properties. DP steels typically have high ultimate tensile strength (UTS) (strengthened by the martensite) combined with low initial yielding stress (enabled by the ferrite), high early-stage strain hardening, and macroscopically homogeneous plastic flow (because of the absence of Lüders effects). These properties make DP steels as one of ideal alloy systems for automotive-related sheet-forming operations. DP steel can be used as engine cross member, hinge reinforcement door, reinforcement shocktower and engine cradle rear cross member [11].

g) CP steel

Karelova said in [12]: CP (Complex Phase) steels provide a higher level of yield strength than DP steels and are therefore an excellent choice for applications where the combination of high strength and ductility together with high energy absorption and high residual deformation capacity is required. Due to the controlled chemical composition and the special manufacturing process, these steels possess a very fine microstructure consisting of ferrite and a higher volume fraction of hard phases (bainite, martensite) than DP steels. CP steel has several automotive applications, particularly in body structure, suspension, and chassis components [4].

h) Martensitic steel

Tamarelli said in [4]: in Martensitic steel (MS), nearly all austenite is transformed to martensite. MS is best known for its extremely high strength, UTS from 900 to 1700

MPa. The martensitic matrix contains a small amount of fine ferrite and/or bainite. Typically this structure forms during a rapid quench following hot-rolling, annealing, or a post-forming heat treatment. MS has relatively low elongation, but ductility can be improved through post-quench tempering obtaining adequate formability considering its extreme strength. It is often used for body structures, ancillary parts, and tubular structures [4]. Quenched and tempered martensitic steels have been used for suspension springs and stabilizers in the automotive industry [13].

i) Maraging steel

Jägle said in [14] : maraging steel has a martensitic matrix that contains a high density of nanometer-sized intermetallic precipitates. The martensitic microstructure is not achieved by carbon, but (usually) by a high amount of nickel. Ni (Al, Fe)-maraging steels with a composition of Fe-18Ni3Al4Mo0.8Nb0.08C0.01B (wt. %) has yield strength 1947 MPa, ultimate tensile strength of 2197 MPa and a total elongation of 8.2% [15].

j) Boron steel

Suski said in [16]: small additions of boron can substantially increase the hardenability of low carbon steels, because of the segregation of boron to the boundaries of austenitic grains. Another theory is that boron can promote bainite formation. Among the steel hardenability elements, boron was regard as the strongest one by far [17].

k) TRIP steel

The microstructure of TRIP (Transformation-Induced Plasticity) steels contains retained austenite embedded in a matrix of ferrite, bainite or martensite depending on alloy composition and heat treatment processing [18] [19]. The mechanical properties of TRIP steels mainly depend on the volume fraction of the metastable retained austenite and its stability against deformation [20]. A typical TRIP steel with

15.8%Mn, 3.0%Si, 2.9%Al and 0.02%C contains ferrite, austenite and martensite [19].

l)   TWIP steel

TWIP (Twinning-induced plasticity) steels obtain their outstanding mechanical properties from the enhancement of the work hardening rate due to the formation of bundles of mechanical nano twins and dislocation glide during straining [21] [22] [23] . A typical TWIP steel with 25.6%Mn, 3.0%Si, 2.8%Al and 0.03%C contains only austenite [19].

Even though steel is the main material used in automotive industry, in order to take advantage of other materials, such as aluminum alloys, composites, the car often contents a mixture of different materials [24]. As an example, the materials in a new Volvo are shown in Fig. 2-3. Ultra high strength steel is used to keep the passenger safe.

Fig. 2-2: Material mix in the new edition Volvo XC90 [25].

Fig. 2-3: Tensile properties of the steels used in Volvo XC90 [26].

As Fig. 2-3 shows, DP steel, HSLA steel, Rephos steel and Boron steel are used in the Volvo XC90, and the definition of HSS (High Stress Steel), EHSS (EHSS Extra high stress steel) and UHSS (UHSS Ultra high stress steel) is also clarified.

a) HSS: yield stress ≥200 MPa; tensile strength ≥340 MPa.

b) EHSS: yield stress ≥280 MPa; tensile strength ≥600 MPa.

c) UHSS: yield stress ≥400 MPa; tensile strength ≥800 MPa.

2.2 High Mn steel

High Mn steel containing 13%Mn was first made by Sir Robert Abbott Hadfield in the 1880s and famous for its high impact strength and resistance to abrasion [27] [28]. Since the 1990s, high Mn steel including TRIP and TWIP steels again got attention from many researchers around the world, such as Georg Frommeyer (MPIE) [19], Wolfgang Bleck (RWTH Aachen University) [29], Dierk Raabe (MPIE) [3], Bruno Charles De Coomann (Pohang University of Science and Technology) [30], Karl-Heinz Spitzer (TU Clausthal) [31], Young-Kook Lee (Yonsei University) [32], and so on.

The excellent mechanical properties of High Mn steel (10-30% Mn) result from different deformation mechanisms, such as crystallographic slip, the martensitic transformation and twinning [33] [34] [35] [36]. In order to reduce the density, Al and Si are added to high Mn steel [37].

2.2.1 Tensile properties at room temperature

In most cases, ductility and strength are tested by the tensile test. From the literature, YS (Yield Stress), UTS (Ultimate Tensile Stress), UE (Uniform Elongation) and TE (Total Elongation) of different high Mn Steels are summarized in the appendix. From Fig. 2-1, made by Max Planck Institute for Iron Research, comparing with other steels, high Mn steel offers the best combination of stress and elongation by far. ArcelorMittal and ThyssenKrupp, two of the main suppliers for the automotive industry, agreed towards the development of light weight and high strength TWIP steels, marketed under the designation X-IP. The elongation to fracture of such materials is approximately 50%, for an ultimate tensile strength of 1000 MPa, or 35% elongation for UTS of 1400 MPa [38]. Fig. 2-4 and Fig. 2-5 were made from the data in [39] [40] [41].

Fig. 2-4: Influence of C on UTS and TE at room temperature (2-3%Si, 2-3%Al, 15%Mn).

Fig. 2-5: Influence of Mn on UTS and TE at room temperature (2-3%Si, 2-3%Al, <0.1%C).

When carbon is below 0.1%, with Mn increasing, the tensile stress decreases, while elongation increases. When Mn is about 15%, carbon has the same effect like manganese, but when carbon content is more than 0.5%, with carbon content increasing, the tensile stress increases slightly.

High Mn TWIP steel exhibit plastic deformation not only by dislocation slip but also by mechanical twinning [42]. The newly formed twin boundaries act as obstacles to dislocation movement to increase tensile stress, in the same way as grain boundaries [43]. That's why high Mn TWIP steel offers a superior combination of strength and ductility.

2.2.2 Energy absorption properties

High Mn TWIP and TRIP steels have better energy absorption than other steels, as Fig. 2-6 shows. This is one important reason why it is a promising material in automotive industry to keep the passenger's safety during collisions [44].

Fig. 2-6: Comparison of the energy absorption, for automotive steels during high strain deformation (Strain rate: $10^3 s^{-1}$) [45].

2.2.3 **Delayed fracture**

Delayed fracture is a phenomenon in which the material suddenly fails under external stress or residual stress [46]. The precise mechanism of delayed fracture is still not known and the complex interaction of factors related to transformation, residual stresses and the influence of hydrogen has made this problem particularly difficult to address. But it is clear that Al added TWIP steels may be considered immune to the problem [30] [47]. The delayed fracture is often tested by deep drawn cups method [48]. Twin boundary in high Mn steel can also be the crack initiation site [49]. The increase of austenite stability is expected to improve the resistance against delayed fracture [50].

2.2.4 **High temperature properties**

To assess the hot ductility of steels, the hot tensile test has been employed as the main research tool [51] [52]. A schematic diagram of the hot tensile test is shown in Fig. 2-7. After the sample has been reheated to the test temperature, tensile test begins until its failure.

Fig. 2-7: Schematic diagram of hot tensile test [53].

Hot ductility is usually researched by a Gleeble apparatus [54].

Fig. 2-8: Hot ductility curves of for high Mn steels at low and 1.5%Al levels [55].

As presented in Fig. 2-8, the reduction of area for 18Mn steel is almost the same from 650 ℃ to 1000 ℃. The explanation from Mintz is "*As the temperature increases, the deterioration in ductility due to grain boundary sliding is approximately balanced by the increased rate of recovery*" [55]. However this is not in accordance with the results in section 4.6.2 of this work.

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The experimental arrangement in   Fig. 2-9 can be used to test as cast samples. Firstly the sample in the silica tube is reheated to the melting point temperature. Secondly when the temperature cools down to the test temperature, the sample is stretched until fracture. In addition, sample can also be compressed by hot compression test. Hot ductility measured in the hot compression test of Fe-9%Mn-0.9%C is better than that of Fe-23%Mn-0.6%C and Fe-16%Mn-0.9%C, which means Mn can make hot ductility of high Mn steel worse [29].

Fig. 2-9: Experimental arrangement of hot tensile test [56].

Temperature also plays a very important effect on hot ductility in the continuous casting. Temperature above 1340 ℃ probably accounts for the formation of all internal cracks and surface longitudinal cracks. Transverse surface cracks in the slab are related to the temperature zone between 700-900 ℃ [57].

Though high Mn steel has so many good properties at room temperature, its hot ductility is still a problem, causing difficulties during continuous casting. Fig. 2-11 shows that high Mn steel (15% Mn, 2.5% Al, 2.5%Si, and 0.7% C) can have cracks during Horizontal Single-Belt Casting (HSBC) in the pilot plant in Clausthal. HSBC process offers many advantages, such as lower operation cost, higher productivity, less energy consumption and a simpler operation, and it has the potential for producing advanced steel grades not easily possible via conventional continuous

casting processes [58]. Details about HSBC in Clausthal, as Fig. 2-10 shows, can be found in [59] [60] [61] [31]. One main goal of this work is to understand the reason why high Mn steel has bad hot ductility and to solve this problem.

Fig. 2-10: Schematic diagram of HSBC in Clausthal [62].

Fig. 2-11: Cracks during coiling (X70MnAlSi 15 2.5 2.5) [63].

For TWIP steel (18%Mn, 0.6%C), total elongation was found to reach a maximum value of 88% at 200 ℃ and then decreased continuously with the increase in test temperature from 300 ℃ up to 600 ℃ [64]. The influence of Nb, V and Ti on high temperature properties of 21Mn high Mn steel was investigated by hot compression tests at 900 ℃, 1000 ℃ and 1100 ℃. The addition of Nb, V and Ti in TWIP steels

generates a slight increase in the peak stress value, and Ti microalloyed TWIP steel exhibits the highest peak stress value [65].

Apart from casting and inline rolling, poor hot ductility is also a major concern in welding processes. Carbon equivalent (CE) is a main factor to evaluate the steel weldability and one empirical formula is as follows [66]:

$$CE = \%C + \frac{\%Mn}{6} + \frac{\%Si}{24} + \frac{\%Cr}{5} + \frac{\%Ni}{40} + \frac{\%Mo}{4} \qquad \text{(eq. 2-1)}$$

The higher the CE value is, the worse the weldability is. If Mn content is higher than 18%, CE is more than 3, which means that high Mn steel is likely to have cracks during welding process. Higher CE in the welded joint leads to higher hardness and hence makes it brittle. C, Mn and Si all lead to bad weldability, as eq. 2-1 shows. The so-called Varestraint test technique has extensively been used for evaluation of weld metal hot cracking susceptibility. Formation of continuous $\gamma + M_3C$ eutectic phase along the grain boundaries mainly influences the hot cracking susceptibility of high manganese steel [67]. In nickel based alloy 718 (18.9% Cr, 18.3% Fe, 2.9% Mo, 4.9% Nb, 0.46% Al, 0.94% Ti, 0.05% C, Ni balance), there is no or very limited deterioration due to the presence of δ-phase [68]. δ-phase can also improve spot weldabilty of TRIP steels by giving better ductility to the weld [69]. High Mn steel hot cracking susceptibility is comparable to that of fully-austenitic Ni-based alloys or special stainless steels. Among high Mn steels, the X70MnAlSi15-3-3 alloy was the most susceptible to hot cracking [70]. Resistance spot welding is usually used in automotive sheet steel welding, and by adopting the constant power control instead of constant current control, the weldability of TWIP steel can be improved by reducing expulsion at the early stage of the welding process [30] [71].

2.2.5 **Phase transformation**

Phase transformation behavior is an important parameter especially for the understanding of high temperature mechanical properties. The Fe-Mn-C phase diagram published by Schumann, Fig. 2-12 has been cited lots of times, but also revised. So the original diagram is reproduced here for reference. Before deformation, if Mn is above 27%, full austenite can be obtained at any carbon content. After deformation γ-phase zone becomes smaller, and the ε-phase zone becomes bigger. But the Al and Si content were not notified by Schumann.

Fig. 2-12: Fe-Mn-C phase diagram before (a) and after (b) steel deformation [72].

From the data in literature [2] [3] [4] [11], Fig. 2-13 was compiled to show the accordant phase diagram of high Mn steel with 2-3%Al and 2-3%Si. After deformation hcp-phase appears in austenite matrix. Mn and C can enlarge the fcc zone, while Al makes it smaller. Si is added mainly to suppress the formation of cementite, but it could cause drawbacks on the steel sheet surface because of Si-Mn oxides formation [73]. In physical metallurgy, elements can be divided into two main groups, called austenite stabilizers and ferrite stabilizers. Mn and C are austenite stabilizers, while Si and Al are ferrite stabilizers [74] [75].

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Fig. 2-13: Fe-Mn-C phase diagram before (left) and after (right) steel deformation with

2-3%Al and 2-3%Si at room temperature (data from [2] [3] [4] [11]).

Al can raise yield stress by solution hardening, suppress cementite precipitation by decreasing both activity and diffusivity of C in austenite, and stabilize austenite against the strain induced ε-martensitic transformation by increasing both Stack Fault Energy (SFE) [76] and matrix strength. Furthermore, the Al addition retards or eliminates hydrogen delayed fracture in Fe-Mn-C TWIP steels [77]. The plasticity mechanisms depend on the SFE. The mechanical martensitic transformation occurs below 18 mJ/m^2 and twinning between 12 and 35 mJ/m^2 [78].

Base on the phase diagram, QP (Quenching and Partitioning) process was invented to control the retained austenite. This process includes quenching austenite below the martensite start temperature, followed by a partitioning treatment to enrich the remaining austenite with carbon, thereby stabilizing it to room temperature [79].

3 **Experimental methods**

In this chapter the performed experimental work is explained. First the preparation of the melt is described. Afterwards shear test is introduced. At last metallography and microsegregation analysis methods are presented.

3.1 **High Mn steel-making**

Experiments with 4 series of steels with 13%, 15%, 17% and 21% Mn were performed. The aimed Al- and Si-content was kept constant being about 2.5% for both. The C-content was varied from about 0.04% to about 0.8% within each series. The corresponding melt of approx. 11kg was made in a vacuum induction furnace (Leybold-Heraeus IS 1.5II), Fig. 3-1. As base charge low alloyed steel was used. The alloying elements Mn, Al, Si and C were added into the steel melt to hit the aimed composition. The composition of the base charge and the alloying agents, (Fig. 3-2), are given in Table 3-2. The density of Al is low, and Al is easily oxidized. So Al is always the last raw material added to the steel melt. The melting and casting was performed in an atmosphere of argon 4.6 (99.996%) at approx. ambient pressure (950 mbar abs.). The crucibles used in the experiments were cast from alumina based concrete (Oxyd Keramik Keroxyd G/A-9h), Table 3-1. After hardening, they were dried at 200 ℃ and afterwards within 10 hours heated up to 1100 ℃ holding for 2 hours. The crucible treated in this way was inserted into the induction coil.

Table 3-1: Composition of crucible (wt. %)

Fe_2O_3	SiO_2	Al_2O_3	TiO_2	CaO	MgO	P_2O_5	MnO	S	K_2O
0.14	0.05	95.50	0.09	2.92	0.28	0.01	0.06	0.09	0.03

Fig. 3-1: Vacuum induction furnace used to prepare high Mn steel.

Table 3-2: Chemical composition of alloy agents (wt. %)

Alloy agents	Fe	C	Si	Mn	Al	Ti	P	S	Cr
Low alloyed Steel	99.7	0.036	0.014	0.19	0.033	0.001	0.003	0.0031	0.015
Graphite	-	99.87	-	-	-	-	-	0.009	-
Si-lumps	0.16		99.6	-	0.19	0.03	0.004	-	-
Mn-lumps	-	0.016	0.25	96.35	-	-	0.005	0.035	-
Al-wire	-		-	-	~100	-	-	-	-

Fig. 3-2: Al, C, Si and Mn alloy agents.

The main processes of steel-making are as follows:

1) Check the water cooling system and the thermocouple used for melt temperature measurement;

2) Fill the low alloyed steel into the crucible;

3) Close the vacuum chamber of furnace, evacuate it to about 0.1mbar, and refill with argon;

4) Turn on the power and melt down the low alloyed steel;

5) Add the alloying agents;

6) Take a sample for on-line chemical analysis using GDOES (Glow Discharge Optical Emission Spectroscopy) spectrometer (LECO GDS850A);

7) Correction of composition;

8) Hold the temperature at about 1550 °C and take the cylinder sample for shear test;

9) Add alloying agents to increase carbon content;

10) Repeat steps 8) and 9);

11) Pour the remaining melt into the mold and turn off the power.

During melt preparation, the temperature was measured continuously by a type B thermocouple (error +/- 0.5%) that was integrated into the crucible. To adjust the accurate melt temperature for the trial, in the final stage the temperature was additionally measured by a second type B thermocouple protected by a quartz tube and inserted directly into the melt.

It has to be noted that, with increasing of experimental duration, some slag formed. In order to get a "clean" sample, the slag was removed occasionally with a hand-guided shovel.

3.2 Shear test

3.2.1 Shear test equipment

High temperature properties of steel are usually investigated by hot torsion test [80] [81] and hot tensile test [82] [83] [84]. The samples used in these tests often are heated from room- to the test-temperature. A disadvantage of this method is that the original solidification structure, which has a predominant influence on the high temperature properties found in casting and in-line rolling, is not present any more. More conclusive in this respect is the Gleeble test, in which the samples are melted in the test apparatus, solidified and cooled to test temperature. But at the same time such tests are also quite challenging e.g. solidification has to be carefully controlled in order to avoid that the results are affected by porosity.

In this work a self-made shear test apparatus was developed and applied, as Fig. 3-3 shows. In these tests the melt is sucked into a quartz tube which is submerged into the melt prepared in the vacuum induction furnace through the vacuum lock. The quartz tube, which is wrapped into an insulating ceramic feld (RHI, Pyrostop 1600), is fixed to a probe holder made from copper. This probe holder is equipped with a type-B thermocouple inside a quartz protection type for continuous temperature measurement

inside the cylinder sample. After approx. 2min holding time allowing for solidification, the quartz tube together with the holder is removed from the furnace. Then the quartz tube is taken off and the solidified cylinder inside is inserted into the shear hole of the shear test machine. When the test temperature is reached, the shear is closed whereby the path length, the force (by the pressure of the hydraulic piston) and the temperature in the cylinder are recorded. This procedure, even though the setup used is still provisionally, has several advantages:

- The diameter of the cylinder sample being 23 mm presents a representative average of the material's structure properties.

- The geometry of the quartz tube enables feeding of melt into the cylinder during solidification, therefore, center porosity is almost avoided and does not lead to a notch effect like very often in tensile tests after melting and resolidification. So, in the shear tests a good reproducibility was found.

- There is no complicated sample preparation required, so the time needed per test is quite short. The steel composition can be varied by alloy additions to the melt. In this way a test series with increasing contents of certain elements can be performed in an efficient way.

- In principal, the solidification and cooling speed can be controlled by insulating the quartz tube (like in this experiments), by gas cooling and by inductive or conductive heating (not available yet in this apparatus yet).

- During the shear test mostly internal cracks are generated in the material close to the sheared surface. As these internal cracks are, in contrast to the shear surface itself, protected against the air, the structure of the crack surfaces is perfectly preserved and can be investigated.

Fig. 3-4 shows a schematic diagram and a photo of the quartz tube and the tip of the probe holder. The probe holder consists of two concentric stainless steel tubes. A cooper screw closes at the cross-section between the two tubes. Between the outer and

inner tube, four plastic tubes lead cooling water close to the cooper screw, in order to avoid it becoming too hot.

Fig. 3-3: Shear test machine with hot cylinder sample.

Fig. 3-4: Schematic diagram of melt sampler.

At first, it was very hard to get a sample free of hole (Fig. 3-5). A suitable procedure and design was developed. It was found that during the sucking process, the furnace must be turned off, in order to interrupt the melt flow and to reach a calm flow state. At first a quartz tube, in which a steel ring was fixed, was used according to Fig. 3-6, but finally it was found that the straight and simple quartz tube was the best choice. In order to test samples with different diameters, a second hole with a smaller diameter of 13 mm was made in the steel shear plate, as shown in Fig. 3-7. Rings from a hard metal were inserted into the shear, whereby the cutting edge has radius of 1 mm.

Fig. 3-5: Cylinder samples after shear test (a hollow sample, b good sample).

Fig. 3-6: Quartz tube with a steel ring inside.

Fig. 3-7: Steel plate of shear test apparatus with small and big holes.

To assess the effect of the initial solidification structure on the mechanical high temperature properties a second test was performed with the lower part of the sample after cooling down to room temperature and subsequent reheating to test temperature.

In this test, the lower part of the as cast sample, was annealed for 1 hour in the muffle furnace (Fig. 3-8), in which a K type thermocouple inserted into a dummy cylinder of similar material and geometry than the test cylinder was used to measure the real temperature. Holding temperature was chosen about 20 ℃ above the test temperature, considering the sample cooling rate of approx. 6 ℃/s during transfer of the sample from muffle furnace to the shear test, which lasts about 3s. It has to be noted that the accuracy of type B and K thermocouple at 1000 ℃ is about ±5 ℃ and ±9 ℃ respectively.

Fig. 3-8: Muffle furnace used in annealing process.

3.2.2 Calculation of elongation in shear test

In a standard tensile test the stress that is tensile force divided by the real or initial cross-section of the sample (e.g. in MPa), is given as a function of elongation that is the ratio of sample length elongation and initial sample length (e.g. in %). For the shear test a corresponding stress and elongation is achieved in the following way.

Fig. 3-9: Schematic diagram used to calculate elongation.

In Fig. 3-9, circle 1 is fixed, and circle 2 is moving from left to right. At the beginning, the two circles are coincident.

AB = x is the moving distance,

OA = R is the radius of sample.

So elongation ϵ can be expressed as follows:

$$\epsilon = \frac{S_{AEBF}}{\pi R^2} \qquad \text{(eq. 3-1)}$$

S_{AEBF} is the area of AEBF and calculated by the following equations:

$$CD = CB = (2 \cdot R - x)/2 \qquad \text{(eq.3-2)}$$

$$OC = OD - CD = x/2 \qquad \text{(eq. 3-3)}$$

$$CE = \sqrt{R^2 - OC^2} = \sqrt{R^2 - \frac{x^2}{4}} \qquad \text{(eq. 3-4)}$$

$$\angle FOE = 2 \cdot \angle COE = 2 \cdot arccos\frac{OC}{OE} = 2 \cdot arccos\frac{x}{2R} \qquad \text{(eq. 3-5)}$$

$$S_{DFCE} = S_{FOED} - S_{EOF} = \frac{arccos\dfrac{x}{2R}}{180} \cdot \pi R^2 - \frac{x}{2} \cdot \sqrt{R^2 - \frac{x^2}{4}} \qquad \text{(eq. 3-6)}$$

$$S_{AEBF} = \pi R^2 - 2 \cdot S_{DFCE}$$

$$= \pi R^2 - \frac{arccos\dfrac{x}{2R}}{90} \cdot \pi R^2 + x \cdot \sqrt{R^2 - \frac{x^2}{4}} \qquad \text{(eq. 3-7)}$$

Finally, elongation can be expressed as follows:

$$\epsilon = \frac{S_{AEBF}}{\pi R^2} = 1 - \frac{arccos\dfrac{x}{2R}}{90} + \frac{x}{\pi R^2} \cdot \sqrt{R^2 - \frac{x^2}{4}} \qquad \text{(eq. 3-8)}$$

3.2.3 **Calculation of shear force and stress**

The driving force of shear test machine is made by the hydraulic pump. Using the law of lever, the force acting on the tested sample was calculated according to Fig. 3-10, Fig. 3-11 and Fig. 3-12. The arrow shows the direction of the force, and fore F_2 is acting on the sample. Engineering shear stress σ_{SE} and True stress σ_{ST} can be calculated from eq. 3-9 to eq. 3-23.

Fig. 3-10 Schematic diagram of the lever used to calculate the force on point B.

Fig. 3-11 Shear force calculation schematic diagram1

Known conditions:

$OA = a = 665$mm, $OB = b = 105$mm, $OC = c = 885mm$, $CA_1 = l_{min} = 570mm$;

cross section area in the hydraulic cylinder $S = 0.00064m^2$.

$$F_1 = P \cdot S \qquad\qquad \text{(eq. 3-9)}$$

.According to the Law of Lever:

$$F_1 \cdot sin\theta \cdot a = F_2/sin\theta \cdot b \qquad\qquad \text{(eq. 3-10)}$$

while the directions of $F_1 \cdot sin\theta$ and $F_2/sin\theta$ are vertical to the lever.

$$sin\theta = \frac{c \cdot sin\gamma}{\sqrt{(c \cdot sin\gamma)^2 + (a - c \cdot cos\gamma)^2}} \qquad\qquad \text{(eq. 3-11)}$$

Fig. 3-12 Shear force calculation schematic diagram 2

$OM = m, HK = l_1, OB = OB_1 = b, HB = l_{max} = 23.9mm$; $BN = n$; $MH =$ $h, BK = l; OK = k$

$$\angle COB = \beta = 48°, \quad \angle B_1MO = 90°, \quad \angle ONB = 90°, \quad \angle MB_1O = \mu = 90° - \beta - \alpha.$$

$$m = b \cdot \sin \mu \tag{eq. 3-12}$$

$$n = l_{max} - m \tag{eq. 3-13}$$

$$MH = ON = \sqrt{b^2 - n^2} \tag{eq. 3-14}$$

$$MB_1 = b \cdot \cos \mu \tag{eq. 3-15}$$

$$HB_1 = MB_1 - MH = b \cdot \cos \mu - \sqrt{b^2 - n^2} \tag{eq. 3-16}$$

$$\frac{HB_1}{MB_1} = \frac{HK}{OM} = \frac{l_1}{m} \tag{eq. 3-17}$$

$$l_1 = \frac{b \cdot \cos \mu - \sqrt{b^2 - n^2}}{b \cdot \cos \mu} \cdot m \tag{eq. 3-18}$$

$$k = OK = \frac{ON}{\cos \angle KON} = \frac{\sqrt{b^2 - n^2}}{\cos\mu} \tag{eq. 3-19}$$

$$\cos\gamma = \frac{b^2 + k^2 - l^2}{2 \cdot b \cdot k} = \frac{21636.06 - (23,84 - x)^2}{21632.1} \tag{eq. 3-20}$$

$$F_2 = \frac{P \cdot 317467}{783225 + \dfrac{\left(665 - \dfrac{11449.91 - (23.84 - x)^2}{24.44}\right)^2}{1 - \left(\dfrac{11449.91 - (23.84 - x)^2}{4328.8}\right)^2}} \ (kN) \tag{eq. 3-21}$$

Unit of pressure P is bar.

$$\sigma_{SE} = \frac{F_2}{\pi R^2} \tag{eq. 3-22}$$

$$\sigma_{ST} = \frac{F}{\pi R^2 - S_{AEBF}} = \frac{\sigma_{SE}}{1 - \epsilon} \tag{eq. 3-23}$$

3.2.4 **Shear test versus tensile test**

Tensile test is the common method to test a material's mechanical properties, and its stress σ_T is along the sample length, while shear stress σ_S is vertical to the sample length direction, as Fig. 3-13 shows.

Fig. 3-13: Schematic diagram of tensile a) and shear b) test.

In order to compare shear test with tensile test at high temperature, an Instron 1195 machine was used to do hot tensile test. Normal structural steel was used, and its composition is presented in Table 3-3.

Table 3-3: Composition of structural steel (wt. %)

Material	Mn	C	Al	Si	Cr	Ni	P	S	Ti
Structural steel	0.64	0.145	0.0095	0.176	0.13	0.198	0.03	0.015	0.0014

Both of tensile and shear test curves can be seen in Fig. 3-14, related detailed parameters is given in Table 3-4. Unlike in the tensile test, when the shear stress reaches maximum, at this time the corresponding elongation is defined as max elongation. Max stress of shear test is much bigger than of tensile test. The reason

may be that the diameters of samples are different. Same results also occurred for high Mn steel in chapter 4.

Fig. 3-14: True shear and tensile stress of structural steel at 800 ℃.

Table 3-4: Comparison of shear test and tensile for structural steel

Test	Sample diameter (mm)	Sample length (mm)	Max stress (MPa)	Max Elongation (%)	Test temperature (℃)
Tensile	6	50	72	31.78	800
Shear	12.8	-	379	30.16	800

3.3 **Metallography**

The steel samples were cut, grinded and polished with water cooling to keep the original microstructure, and nital was used for etching. CamScan 44 (EDX, Energy Dispersive X-ray spectroscopy) and Cameca SX100 (WDS, Wavelength Dispersive X-ray spectroscopy) were used to analyze the composition of different phases in the steel. Ferrite content (in volume %) was analyzed by FERITSCOPE FMP30

according to the magnetic induction method, which is usually used in the welding field.

3.4 Microsegregation analysis

Microsegregation plays an important role for high temperature mechanical properties and cracking susceptibility and has been, therefore, analyzed.

3.4.1 Microsegregation ratio

Microsegregation can lead to cracks and lower mechanical properties [85]. The microsegregation ratio method is used in this thesis.

$$\varphi_i = \frac{x_i}{\frac{1}{n}\sum_1^n x_i} \qquad \text{(eq. 3-24)}$$

φ_i Microsegregation ratio

x_i Composition data (wt. %) at a certain point

n Total number of data points

$\frac{1}{n}\sum_1^n x_i$ Average composition

In order to get the maximum and minimum microsegregation ratio (φ_{max}, φ_{min}), at least two microsegregation zones were covered in the line scan. The advantage of this method is that it is very practical and easy to understand. It should be noticed that the line scan which can have hundreds of data points (256 in this thesis) is very necessary to analyze the microsegregation, because the matrix composition of the sample has probably not the same chemical composition which was analyzed by chemical analysis. Making the average for local composition data to get the base composition is a good method instead of using the results from global chemical analysis.

3.4.2 **Partition coefficient**

Scheil equation has been often used to analyze microsegregation during solidification and it is the most simple and powerful tool compared to other solidification models, such as Brody and Fleming model. The most amazing aspect is that this equation can be totally derived with math. The hatched areas in Fig. 3-15 are equal.

$$(C_L - C_s)df_s = (f_L)dC_L \qquad \text{(eq. 3-25)}$$

So the following equation can be derived.

$$C_s = kC_0(1 - f_s)^{k-1} \qquad \text{(eq. 3-26)}$$

Where C_s is the composition of the solid phase, C_0 is the nominal alloy composition, f_s is the fraction of solid, and k is the partition coefficient.

$$k = \frac{C_s}{C_L} \qquad \text{(eq. 3-27)}$$

Fig. 3-15: Solidification scheme used for deriving the Scheil equation [86].

The equilibrium partition coefficient k is a critical physical parameter in studies of solute redistribution during solidification in such processes as metal casting, zone refining, and crystal growth. The simplest and most straightforward method is to

utilize liquidus and solidus curves directly from a phase diagram [87] [88]. But equations of these curves are rarely given in the literature, especially for high Mn steels with Al, Si, and C.

Another way to calculate k is using the experimental data from lining scanning microsegregation zone. However, it is difficult to put the experimental data to the right position of solid fraction. Traditional method is that the experimental data is assumed to be distributed evenly between $f_s = 0$ and $f_s = 1$. But all the data is measured at room temperature, instead of during the solidification at high temperature. Therefore a new method to calculate k is used in this thesis.

$$Q = \sum_i (C_i - \overline{C_i})^2 \qquad \text{(eq.3-28)}$$

C_i experimental data equidistant along the line scan, corresponding $\overline{C_i}$ scheil equation value.

According to the least squares method, Q takes its minimum for the best guess of partition coefficient k which can be calculated using Matlab program [89]. Unlike Mn, Si and C, partition coefficients of Al and Cr [90] are more than 1.

4 Experimental results

High temperature shear tests were performed for 13Mn, 15Mn, 17Mn and 21Mn steels. Temperature, shear stress and elongation for each sample were measured. EDX line scans were done in order to analyze the microsegregation. Ferrite content in the sample was also tested.

4.1 Temperature measurement

In this work, the steels containing 13%, 15%, 17%, and 21%Mn are named as 13Mn, 15Mn, 17Mn and 21Mn respectively. All the shear tests for those samples were performed at 1000 ℃.

Fig. 4-1: Continuous temperature measurement during shear test.

As Fig. 4-1 shows, temperature curves at 1000 ℃ fluctuate a little, because of shear test interference. The sample designation 13-0.07 means that this sample has 13% Mn and 0.07% carbon. Temperature of the sample is measured during the whole process. The maximal temperature of sample 13-0.68 is lower than that of 13-0.07, because with carbon content increasing, the melting point of the sample is decreasing, therefor, a lower superheat is chosen for the melt. From this, it can be also assumed that the thermocouple in the sample worked very well and reliable. When a set temperature is reached, the sample is taken out of crucible and the furnace and is transferred to the

shear test device. There the fiber insulation and the quartz tube is removed, see Fig. 3-3 and Fig. 3-4. Then, according to the cooling curves, cooling rate is about 6 ℃/s is achieved. When the test temperature is reached (1000 ℃ in these experiments) the sample is introduced into the shear and the test is performed. The temperature the test is performed usually reflects in some irregularities of the temperature profile, Fig. 4-1, which serves as a control for the correct test temperature.

4.2 **Elongation speed measurement**

The high temperature mechanical properties depend also on the elongation rate. Fig. 4-2 a shows the shear displacement and Fig. 4-2 b the elongation according to（eq. 3-8） as a function of time for a typical trial (13-0.13-as-cast). From this we get an average displacement speed of $\dot{x} = 8.7 mm/s$ and elongation speed $\dot{\varepsilon} = 39.1\%/s$

Fig. 4-2 Displacement and elongation speed in the shear test

4.3 **Results for 13Mn**

4.3.1 **Chemical composition**

Chemical composition of 13Mn is presented in Table 4-1, the average values of Mn, Al and Si are 13.0%, 2.76% and 2.92% respectively, and carbon content is from 0.07% to 0.68%.

Table 4-1: Chemical composition (wt. %) of 13Mn steels with different C contents

Sample	Mn	C	Al	Si	Cr	Ni	P	S	Ti
13-0.07	13.1	0.068	2.76	2.98	0.072	0.025	0.009	0.010	0.005
13-0.15	13.0	0.149	2.79	2.93	0.062	0.025	0.006	0.009	0.001
13-0.26	13.0	0.264	2.90	2.95	0.080	0.027	0.015	0.008	0.013
13-0.30	12.9	0.303	2.75	2.92	0.056	0.024	0.008	0.009	0.000
13-0.43	13.1	0.429	2.76	2.87	0.059	0.024	0.010	0.008	0.000
13-0.51	13.1	0.510	2.71	2.89	0.063	0.023	0.011	0.009	0.002
13-0.59	13.1	0.589	2.70	2.91	0.051	0.024	0.007	0.010	0.000
13-0.68	13.0	0.675	2.72	2.88	0.099	0.042	0.012	0.009	0.001
Average	13.0	-	2.76	2.92	0.068	0.027	0.010	0.009	0.003

4.3.2 **Results for visual cracking tendency**

The surfaces of high Mn steels with different carbon contents in the as cast state after shear test at 1000 ℃ can be seen in Fig. 4-3. The annealed samples in contrast have no cracks, as shown in Fig. 4-4.

| 13-0.07 | 13-0.15 | 13-0.26 | 13-0.30 |

| 13-0.43 | 13-0.51 | 13-0.59 | 13-0.68 |

Fig. 4-3: Surfaces of as cast 13Mn steels with different carbon contents.

| 13-0.07 | 13-0.15 | 13-0.26 | 13-0.30 |

| 13-0.43 | 13-0.51 | 13-0.59 | 13-0.68 |

Fig. 4-4: Surfaces of annealed 13Mn steels with different carbon contents.

The samples 13-0.07-as-cast up to 13-0.30-as-cast show a rough shear surface while those of 13-0.42-as-cast up to 13-0.59-as-cast are much smoother. A certain roughness is also found for 13-0.07-annealed and 13-0.30-annealed. The shear surface of the other annealed samples is smooth. Slight cracks on the cylinder surface are found for 13-0.30-as-cast up to 13-0.59-as-cast. 13-0.68-as-cast shows strong cracks. The annealed samples are free of cracks on cylinder surface.

4.3.3 Results for shear stress

Just like the tensile test, shear stress curves can also be obtained using the method described in sections 3.2.2 and 3.2.3. Both engineering shear stress and true stress are presented in Fig. 4-5 and Fig. 4-6.

Fig. 4-5: Engineering shear test curves of 13Mn steels at 1000 ℃

Fig. 4-6: True shear stress curves of 13Mn steels at 1000 ℃.

After reaching the maximum the strength decreases linearly close to zero for the annealed samples and for 13-0.43-as cast and 13-51-as cast. For 13-0.07-as-cast up to 13-0.30-as-cast the linear decrease stops at a certain level and there is a more flat wavy profile afterwards which even may go up again. This may be explained by a stick-slip behavior which reflects in the rough shear surface. 13-0.59-as-cast shows a slight and 13-0.68-as-cast strong abnormal behavior which corresponds with the crack formation on the cylinder surface. As will be explained later in more details the stick-slip behavior corresponds with δ/γ two phase structure.

4.3.4 Results for cross section microstructure

The diameter of the samples in Fig. 4-7 is 23 mm. It can be seen that the samples are solid without a larger shrinkage hole in the center. There is a little porosity in the sample 13-0.07. The top dark area in the sample 13-0.68 is caused by the cracks on the sample surface. The darker areas in the cross-section of 13-0.07-as-cast are due to ferrite phase fraction visible after the performed Nital-etching.

Fig. 4-7: Cross section microstructures of 13Mn as cast samples.

Table 4-2 Shows the results for the ferrite fraction measurement (volume, %) achieved with the Ferriscope. Ferrite content is reduced by annealing treatment.

Table 4-2. Ferrite fraction in 13Mn steels.

	13-0.07	13-0.15	13-0.26	13-0.30	13-0.43	13-0.51	13-0.59	13-0.68
As cast	56.70	31.46	21.72	11.38	5.26	2.14	0.17	0
Annealed	42.56	19.06	8.76	2.02	0.56	0.15	0	0

4.3.5 **Results for EDX line scans**

The state of microsegregation is investigated by SEM-analysis combined with concentration measurements of Mn, Al and Si using the line scan technique. Fig. 4-8 shows for the as cast SEM photos of areas from the cylindrical sample cross-section. The radius from the areas is about 6 mm. The red lines mark the path of the line scans. The corresponding concentration results in terms of the segregation ratio are given in Fig. 4-9.

For 13-0.07-as-cast up to 13-0.26-as-cast all segregation peaks are characterized by a reduced Mn-, an increased Si- and an increased Al-content. As will be explained later in more detail this type of segregation occurs during the $\delta \rightarrow \gamma$ transformation. This transformation does not take place completely resulting in a two phase δ/γ structure. The δ is characterized by the reduced Mn-concentration and it appears darker in the SEM photo (due to the higher electrical conductivity of the δ). As the diffusion velocity is small below 1000 °C, it can be assumed that the segregation profile analyzed at room temperature is only very little different from that at testing temperature (1000 °C). The primary segregation due to $l \rightarrow \gamma$ transformation is no more visible due to the large diffusion coefficients in the δ and the resulting homogenization. For 13-0.30-as-cast a second type of segregation appears characterized by an increase of Mn- and Si-concentration and a decrease of

Al-concentration. This type of segregation peaks are located inbetween the peaks from $\delta \rightarrow \gamma$ transformation and have their origin from $l \rightarrow \delta/\gamma$ transformation. From the SEM photo it can be seen that the ferrite phase fraction is decreased and clear stripe morphology found for 13-0.07-as-cast up to 13-0.26-as-cast starts to fade. For 13-0.43-as-cast The $l \rightarrow \delta/\gamma$ transformation induced segregation becomes the predominate one and there is only a slight $\delta \rightarrow \gamma$ transformation induced segregation. For 13-0.51-as-cast, 13-0.59-as-cast and 13-0.68-as-cast there is only the $l \rightarrow \gamma$ transformation which is characterized by a strong positive Si-segregation. After annealing (1h at 1020 ℃) the general behavior is same. The segregation level for the $\delta \rightarrow \gamma$ transformation induced segregation is still the same because it is determined by the δ/γ distribution coefficients but the δ phase fraction seems to be reduced. In contrast the $l \rightarrow \delta/\gamma$ transformation induced segregation ratio is considerably reduced e.g. for Si from ~2.0 in the as cast state to 1.4 after annealing.

Fig. 4-8: Microstructures of EDX line scanning areas for 13Mn as cast samples.

Fig. 4-9: Line scans of 13Mn as cast samples.

Fig. 4-10: Microstructures of EDX line scanning areas for 13Mn annealed samples.

Fig. 4-11: Line scans of 13Mn annealed samples.

4.4 **Results for 15Mn**

4.4.1 **Chemical composition**

Chemical composition of 15Mn is presented in Table 4-3. The average value of Mn, Al and Si are 15.51%, 2.26% and 2.34%, respectively and carbon content is from 0.13% to 0.78%. Sample 15-0.46 has 15.2%Mn, but sample 15-0.69 has 15.72%Mn. About 5g steel was drilled down from the sample for the chemical analysis. During the steel making process, in order to increase carbon content by 0.1% each time, carbon and small amount of Si Al Mn were added into the melt.

Table 4-3: Chemical composition (wt. %) of 15Mn steels with different C contents

Sample	Mn	C	Al	Si	Cr	Ni	P	Se	S	Ti
15-0.13	15.52	0.126	2.24	2.32	0.022	0.024	0.012	0.008	0.047	0.021
15-0.20	15.38	0.197	2.31	2.36	0.021	0.025	0.008	0.008	0.050	0.010
15-0.30	15.65	0.303	2.25	2.42	0.023	0.023	0.008	0.008	0.036	0.011
15-0.38	15.43	0.383	2.26	2.33	0.026	0.022	0.007	0.008	0.042	0.010
15-0.46	15.2	0.462	2.26	2.24	0.029	0.021	0.005	0.007	0.048	0.008
15-0.69	15.72	0.685	2.22	2.37	0.031	0.021	0.006	0.008	0.044	0.011
15-0.78	15.65	0.783	2.26	2.35	0.038	0.026	0.004	0.008	0.066	0.012
Average	15.51	-	2.26	2.34	0.03	0.02	0.01	0.01	0.05	0.01

4.4.2 **Results for visual cracking tendency**

From sample 15-0.38-as-cast on, the samples begin to have cracks on the cylindrical surface (Fig. 4-12), and all the shear surfaces are rough. After annealing all the samples have no cracks on the cylindrical surface, even on the sample with 0.78% carbon. The shear surfaces after annealing are relatively smooth.

| 15-0.13 | 15-0.20 | 15-0.30 | 15-0.38 |

| 15-0.46 | 15-0.69 | 15-0.78 |

Fig. 4-12: Surfaces of as cast 15Mn steel with different carbon contents.

15-0.13   15-0.30   15-0.38

15-0.46   15-0.69   15-0.78

Fig. 4-13: Surfaces of annealed 15Mn steel with different carbon contents.

4.4.3 **Results for shear stress**

It can be seen from Fig. 4-14 and Fig. 4-15 that in the as cast state all profiles show the wavy nonlinear decrease after reaching the maximum stress indicating a stick-slip behavior. This corresponds to the rough shear surfaces found for the as cast state. After annealing there is no stick-slip tendency anymore and the max-stress at the elongation at max-stress are considerable larger. During the sampling and shear test process, due to the thermocouple working not properly, sample 15-0.78 was not sheared. Because the lower part of samples 15-0.20 and 15-0.38 is too short, they were not annealed for the shear test.

Fig. 4-14: Engineering shear test curves of 15Mn steels at 1000 ℃.

Fig. 4-15: True shear stress curves of 15Mn steels at 1000 ℃.

4.4.4 **Results for cross section microstructure**

It can be seen in Fig. 4-16 that there is no macroscopic, visible porosity. The sample with high carbon has transgranular microstructure, which is very detrimental to the hot ductility. As can be seen

Fig. 4-16: Cross section microstructures of 15Mn as cast samples.

Table 4-4 Shows the results for the ferrite fraction measurement (volume, %) achieved with the Ferriscope. Ferrite content is reduced by annealing treatment.

Table 4-4. Ferrite fraction in 15Mn steels.

	15-0.13	15-0.20	15-0.30	15-0.38	15-0.46	15-0.69	15-0.78
As cast	17.26	8.58	4.88	2.82	0	0	0
Annealed	1.98	-	0.30	0	0	0	0

4.4.5 Results for EDX line scans

As can be seen from Fig. 4-17, Fig. 4-18 , Fig. 4-19 and Fig. 4-20, the obvious microsegregation begins to appear in the sample 15-0.46 for the as cast state and in sample 15-0.20 for the annealed state. After annealing, the ferrite phase becomes smaller and less.

Fig. 4-17: Microstructures of EDX line scanning areas for 15Mn as cast samples.

Fig. 4-18: Line scans of 15Mn as cast samples.

Fig. 4-19: Microstructures of EDX line scanning areas for 15Mn annealed samples.

Fig. 4-20: Line scans of 15Mn annealed samples.

4.5 **Results for 17Mn**

4.5.1 **Chemical composition**

Chemical composition of 17%Mn is presented in Table 4-5. The average values of Mn, Al and Si are 17.21%, 2.40% and 2.28%, respectively, and carbon is from 0.08% to 0.72%.

Table 4-5: Chemical composition (wt. %) of 17Mn steels with different C contents

Sample	Mn	C	Al	Si	Cr	Ni	P	Se	S	Ti
17-0.08	17.52	0.084	2.40	2.28	0.029	0.023	0.005	0.009	0.052	0.030
17-0.17	17.91	0.168	2.42	2.27	0.026	0.022	0.005	0.010	0.039	0.032
17-0.35	17.28	0.345	2.40	2.30	0.029	0.022	0.005	0.009	0.078	0.027
17-0.44	17.21	0.442	2.41	2.19	0.028	0.023	0.004	0.009	0.056	0.027
17-0.51	17.29	0.512	2.413	2.235	0.032	0.024	0.005	0.009	0.038	0.028
17-0.58	17.52	0.582	2.40	2.28	0.029	0.023	0.005	0.009	0.052	0.030
17-0.65	17.64	0.651	2.31	2.351	0.029	0.022	0.005	0.009	0.037	0.031
17-0.72	17.81	0.721	2.42	2.31	0.028	0.023	0.005	0.010	0.063	0.032
Average	17.52	-	2.40	2.28	0.029	0.023	0.005	0.009	0.052	0.030

4.5.2 **Results for visual cracking tendency**

17Mn as cast samples began to have cracks at 0.35% carbon, as presented in Fig. 4-21. Compared to13Mn and 15Mn steels, 17Mn have cracks at lower carbon content,

which suggests that Mn also can make hot ductility worse. All the samples after annealing showed no cracks (Fig. 4-22).

| 17-0.08 | 17-0.17 | 17-0.35 | 17-0.44 |

| 17-0.51 | 17-0.58 | 17-0.65 |

Fig. 4-21: Surfaces of as cast 17 Mn steel with different carbon contents.

| 17-0.17 | 17-0.35 | 17-0.44 |

| 17-0.51 | 17-0.72 |

Fig. 4-22: Surfaces of annealed 17 Mn steel with different carbon contents.

4.5.3 **Results for shear stress**

As can be seen from Fig. 4-23 and Fig. 4-24, the results for 17Mn are similar to those for 13Mn. The detailed influence of C on stress and elongation will be discussed in the next chapter.

Fig. 4-23: Engineering shear test curves of 17Mn steels at 1000 ℃.

Fig. 4-24: True shear stress curves of 17Mn steels at 1000 ℃.

4.5.4 **Results for cross section microstructure**

It can be seen from Fig. 4-25 that the grain size of sample 17-0.72 is much larger than for sample 17-0.08.

Fig. 4-25: Cross section microstructures of 17Mn as cast samples.

Table 4-6 Shows the results for the ferrite fraction measurement (volume, %) achieved with the Ferriscope. Ferrite content is reduced by annealing treatment.

Table 4-6. Ferrite fraction in 17Mn steels.

	17-0.08	17-0.17	17-0.35	17-0.44	17-0.51	17-0.58	17-0.65
As cast	9.65	3.36	0.29	0	0	0	0
Annealed	0.482	0	0	0	0	0	0

4.5.5 **Results for EDX line scans**

Fig. 4-26: Microstructures of EDX line scanning areas for 17Mn as cast samples.

Fig. 4-27: Line scans of 17Mn as cast samples.

Fig. 4-28: Microstructures of EDX line scanning areas for 17Mn annealed samples.

Fig. 4-29: Line scans of 17Mn annealed samples.

From Fig. 4-26 and Fig. 4-27, serious microsegregation begins at 0.35% carbon in as cast state, and Si microsegregation ratio in 17-0.35 is very high, and the maximum value is 3.2. As presented in Fig. 4-28 and Fig. 4-29, the microsegregation ratio after annealing is much lower than in as cast state.

4.6 Results for 21Mn

4.6.1 Chemical composition

Chemical composition of 21Mn is given in Table 4-7, the average value of Mn, Al and Si are 21.31%, 2.33% and 2.44% respectively, and carbon content varies from 0.10% to 0.80%.

Table 4-7: Chemical composition (wt. %) of 21Mn steels with different C contents

Sample	Mn	C	Al	Si	Cr	Ni	P	Se	S	Ti
21-0.10	20.91	0.104	2.20	2.21	0.027	0.022	0.005	0.012	0.029	0.021
21-0.16	21.38	0.158	2.3	2.56	0.028	0.020	0.005	0.011	0.038	0.015
21-0.22	20.91	0.219	2.67	2.44	0.033	0.022	0.005	0.010	0.055	0.014
21-0.32	21.78	0.322	2.33	2.56	0.028	0.021	0.005	0.011	0.070	0.016
21-0.43	21.04	0.425	2.23	2.27	0.027	0.021	0.004	0.012	0.030	0.021
21-0.55	21.83	0.549	2.24	2.59	0.026	0.021	0.004	0.011	0.048	0.015
21-0.67	21.31	0.673	2.33	2.44	0.028	0.021	0.005	0.011	0.045	0.017
21-0.80	21.31	0.797	2.33	2.44	0.028	0.021	0.005	0.011	0.045	0.017
Average	21.31	-	2.33	2.44	0.028	0.021	0.005	0.011	0.047	0.017

4.6.2 **Results for visual cracking tendency**

It can be seen in Fig. 4-30 that, except for the first sample 21-0.10, all samples have cracks. In contrast to this, the 13Mn steels only showed cracks for the last sample 13-0.68 with the highest carbon content. The hot ductility of 21Mn is very poor, because of its high Mn content. It can be assumed the steel containing more than 21% is very difficult to cast in the steel plant.

The samples after annealing have better hot ductility. But annealed samples 21-0.43 and 21-0.55 still show some little cracks on the surface. This means that when the Mn content is very high, cracking is inevitable even after 1 hour annealing.

| 21-0.10 | 21-0.16 | 21-0.22 | 21-0.32 |

| 21-0.43 | 21-0.55 | 21-0.67 | 21-0.80 |

Fig. 4-30: Surfaces of as cast 21Mn steel with different carbon contents.

21-0.16   21-0.22   21-0.32

21-0.43   21-0.55

Fig. 4-31: Surfaces of annealed 21Mn steel with different carbon contents.

4.6.3 Results for shear stress

From Fig. 4-32 and Fig. 4-33, it can be assumed that the samples with cracks may nevertheless have good elongation which is not equal to a good ductility. In addition, the true stress curve of as cast sample 21-0.80 has two peaks. It is assumed that the rupture occurred at the first peak.

Fig. 4-32: Engineering shear test curves of 21Mn steels at 1000 ℃.

Fig. 4-33: True shear stress curves of 21Mn steels at 1000 ℃.

4.6.4 **Results for cross section microstructure**

The sample diameter in Fig. 4-34 is 23 mm. In the sample 21-0.80, the columnar crystals structure is very obvious. It can be assumed that in full austenite steel, austenite microstructure would be grow very long, because there is no obstacle during the growing process. While in ferritic+austenitic sample, ferrite is the obstacle to stop austenite growing.

Fig. 4-34: Cross section microstructures of 21Mn as cast samples.

Table 4-8 Shows the results for the ferrite fraction measurement (volume, %) achieved with the Ferriscope. After annealing, all the 21Mn samples have no ferrite any more.

Table 4-8. Ferrite fraction in 21Mn steels.

	21-0.10	21-0.16	21-0.22	21-0.32	21-0.43	21-0.55	21-0.67	21-0.80
As cast	1.86	1.05	0.23	0	0	0	0	0
Annealed	0	0	0	0	0	0	0	0

4.6.5 Results for EDX line scans

As can be seen from Fig. 4-35 and Fig. 4-36, the obvious microsegregation appears at 0.22% carbon. Si microsegregation ratio in sample 21-0.22 and 21-0.32 is high, and the value is approx. 2.5. It can be seen from Fig. 4-37 and Fig. 4-38 that microsegregation ratio after annealing is low.

Fig. 4-35: Microstructures of EDX line scanning areas for 21Mn as cast samples.

Fig. 4-36: Line scans of 21Mn as cast samples.

Fig. 4-37: Microstructures of EDX line scanning areas for 21Mn annealed samples.

Fig. 4-38: Line scans of 21Mn annealed samples.

4.7 Results for 15Mn from belt strip casting

4.7.1 Chemical composition

In order to test the temperature effect on stress and elongation, samples cut from HSBC strip were used. The original thickness of the strip is 15 mm. The diameter of samples in Fig. 4-39 is 12.8 mm. A notch was made on the top to show the original

orientation position of the sample in the strip. Table 4-9 gives the chemical composition of this steel.

Table 4-9: Composition of steels from HSBC (wt. %)

Sample	Mn	C	Al	Si	Cr	Ni	P	S	Ti
15-0.85	14.9	0.85	2.71	2.63	0.0643	0.031	0.0139	0.0104	0.0219

Fig. 4-39: High Mn steel samples from HSBC.

4.7.2 **Results for shear stress**

After annealing in the muffle furnace for 1 hour at 1020 ℃, shear test were done at 800, 900 and 1000 ℃. The sample designation "800-Vertical" means that shear test was done at 800 ℃ and shear test direction is vertical to the strip, as Fig. 4-40 shows.

Fig. 4-40: Shear test direction

As presented in Fig. 4-41 and Fig. 4-42, shear stress at 800 ℃ is much higher than at 900 ℃ and 1000 ℃, but shear stresses at 900 ℃ and 1000 ℃ are almost the same, except for the samples 900-Vertical and 1000-Vertical. Furthermore, shear stress tested from horizontal direction is higher than from vertical direction. The possible reason is that during the casting process, the cooling rate and heat flux at top and bottom of strip are much higher than at the strip's sides, which leads to the grains in the microstructure grow in the vertical direction.

Fig. 4-41: Engineering shear stress of high Mn steels from HSBC.

Fig. 4-42: True shear stress of high Mn steels from HSBC.

Comparing the stress levels for the 1000 ℃ test temperature with the results for 15-0.78-annealed they are similar whereby the ductility (elongation at max stress) is lower. In this respect the influence of the lower sample diameter has to be considered. The first part of curves for 900 ℃ and 1000 ℃ both for horizontal and vertical samples are similar. The differences in the max stress and elongation may be due to material defects in the as cast structure. This may also be the explanation for the anisotropy between the horizontal and vertical direction.

5 Discussion

Based on the experimental results, the following aspects are discussed. In section 5.1 the cracking susceptibility achieved by visual inspection is summarized and discussed. Microstructure in the crack and precipitation in it was analyzed in section 5.2. Section 5.3 deals with the influence of carbon and ferrite content on the shear stress and elongation. Three types of solidification of high Mn steel were found in section 5.4. In section 5.5, microsegregation was analyzed and discussed.

5.1 Cracking susceptibility and industrial production proposal

The main purpose of the experiments is to find a proper alloy with good hot ductility. As can be seen in Fig. 5-1, the red dots alloys had large severe cracks in high temperature shear test on the cylindrical surface according to the visual inspection, and thus the hot ductility is poor. The green dot alloys had no cracks in high temperature shear test and the yellow only small minor cracks.

(a)

Fig. 5-1: Cracking tendency of high Mn steel before (a) and after (b) annealing.

The alloys in Fig. 5-1 (a) show the as cast state and Fig. 5-1 (b) the cracking susceptibility after annealing. Some as-cast cracking samples, such as 13-0.68,

21-0.22, etc. exhibiting cracks in the as cast state, after annealing, had no cracks in the 1000 ℃ shear test. This indicates that the hot ductility of high Mn alloy will increase much better after annealing.

(a)   (b)

Fig. 5-2: Phase of high Mn steel before (a) and after (b) annealing.

In Fig. 5-2, the influence of chemical composition on the occurring phases is shown. The red dots alloys had full fcc austenite microstructure, while the green dots alloys had ferritic+austenitic microstructure. After annealing at 1020℃ for 1 hour, the ferritic + austenitic alloys, such as 13-0.59, 21-0.10 etc., turned into a full austenitic microstructure. The classification is based on the measurement with the ferriscope.

Based on the above experimental results and analysis, in order to give a direct hint which high Mn steel can be casted in industrial production, Fig. 5-3 was made.

Fig. 5-3: Hot ductility and microstructure phases of high Mn steel.

The as cast high manganese steel alloys below the black line (lower right part coinciding with blue line) in Fig. 5-3 had no cracks during the 1000 ℃ high temperature shear test. The alloys below the red line had no cracks in the shear test after annealing at 1020 ℃ for 1 hour, while those above the red line had cracks (including the red line). The alloys below the blue line consist of ferrite + austenite and these above are austenitic. The alloys below the olive line after annealing have a ferritic + austenitic microstructure, while those above the line are fully austenitic.

The zone below the black line and the zone below the blue line are not coincident in any case, which indicates that it is impossible to produce as cast fully austenitic high Mn steel without cracks. But the zone below the black line and the zone above the olive line have a coincidence zone, which is designated as A in the figure. That means these alloys in zone A can be casted without cracks in the continuous casting process and have fully austenitic microstructure after annealing. If the annealing time increases, zone A should become wider. Hence, alloys in zone A can be chosen to produce full austenite high Mn steels both for conventional continuous casting and HSBC.

5.2 **Precipitation in the cracks**

Of course, it is well known that apart from ferrite precipitation also the precipitation of nitrides, carbides and sulfites lead to reduction of ductility especially at high temperatures. In a tensile test at high temperatures the crack surface oxidises and the original surface structure is destroyed. So, it is difficult to identify precipitates on the crack surface. In comparision to the hot tensile test, cracks can be observed much better after hot shear test. In shear test internal cracks are generated below the shear surface which are protected against oxidation and reveal the original situation at crack formation.

Fig. 5-4: Macro- and microstructure of cracks at 13-0.68-as-cast.

The macrostructure and microstructure of a typical internal crack for sample 13-0.68-as-cast can be seen in Fig. 5-4. This sample shows a especially large difference both for stress and ductility between as cast and annealed state. Furthermore, the as cast data do not follow the general trend in the 13Mn series, like

those for 15-0.46-as-cast and 15-0.69-as-cast in the 15Mn series, see Fig. 5-13. In Fig. 5-4 two types of crack surfaces are seen. One type, Fig. 5-4c has an overall smooth appearance with a high density of small craters. The precipitates in the center of these craters are too small to be analysed by the equipment available in this investigation. But it is known that such types of craters are often originated by manganesesulfides. The other type of surfaces, Fig. 5-4 e, f is characterized by precipitates with a dendritic structure, whereby the arm distances are much smaler then for the solidification structure. The increased Ti- and at some points also Nb-content, Table 5-1, suggests that this structure is formed by carbides.

Table 5-1 Composition of matrix and needle structure in the crack

Elements	1	2	3	4	5
Fe	66.45	56.31	38.06	49.67	61.69
Mn	19.38	13.65	14.25	16.08	18.32
Si	8.82	6.03	3.77	6.49	7.33
Al	4.24	3.73	1.53	3.07	3.68
S	0	0.05	0.20	0.17	0.04
Ti	1.12	3.86	29.87	12.70	1.72
O	0	16.37	10.38	10.02	6.99
Nb	0	0	1.39	1.13	0.04
Cr	0	0	0.56	0.29	0.11
P	0	0	0	0.36	0.09

After annealing, the volume of carbides becomes much less [91]. That is one reason why the samples after annealing have a significantly improved ductility. Internal cracks at several locations for 15-0.78-as-cast are shown in Fig. 5-5. In most cases the crack surfaces has a shaggy structure with quite regular cells, Fig. 5-5 a, c, e, f. The surface in Fig. 5-5d is smooth and might have been polished by friction between the upper and lower crack surface. The scale of the cellular structure is different reaching from a type of dendrite arms, Fig. 5-5e, to a very fine cellular structure in the area around point 5 and 6 in Fig. 5-5f. At the points marked in the figure the chemical composition has been measured by EDX. Apart from point 1, 2 and 9 at all other points the Mn-content and the Si-content is increased and the Si-content is mostly considerable lager as the Al-content. Furthermore, the P-content is sometime increased too. This composition is typical for the final solidification zone of this grade, Fig. 4-18.

The points 1 and 2 have a composition which is typical for a zone inbetween to final solidification zones. That means the Mn-content is lower and the Al-content is larger than the Si-content. Point 9 has an increased Ti- and Nb-content characterizing a carbide. A significant density of precipitates was not found on the crack surface of this sample. Furthermore, the reason for the cellular morphology of the crack surfaces could not been found with the equipment available in this investigation. It may be that the crack surfaces are more or less final solidification areas where the yield strength is reduced by the change in chemical composition. It is also possible that there is a cementite structure below the cells formed in the zones affected by microsegregation, see Fig. 5-5a point 9, 10. The reduced C-content beneath the cementite may lead to an adjacent ferrite layer with a lower yield strength than the bulk austenite. It should be mentioned that a test with NdFeB-magnet reveals the presence of ferrite also in this material at room temperature.

Fig. 5-5: Macro- and microstructure of cracks at 15-0.78-as-cast.

Table 5-2: Chemical composition measured at the marked points in Fig. 5-5.

	Fe	Mn	Si	P	Al	Ti	Cu	N	C	Nb	O
1	74.36	17.38	3.86	0.20	4.21	0	0	0	0	0	0
2	75.82	15.67	3.88	0.17	4.46	0	0	0	0	0	0
3	67.95	22.50	6.10	0	3.45	0	0	0	0	0	0
4	68.04	22.52	6.07	0.06	3.21	0	0	0	0	0	0
5	66.85	23.03	6.58	0.22	3.31	0	0	0	0	0	0
6	67.58	22.30	6.73	0	3.39	0	0	0	0	0	0
7	69.68	20.30	5.87	0	4.14	0	0	0	0	0	0
8	67.08	23.07	6.42	0.06	3.37	0	0	0	0	0	0

continued	Fe	Mn	Si	P	Al	Ti	Cu	N	C	Nb	O
9	58.39	22.45	4.33	0.45	1.80	10.58	0	0	0	1.99	0
10	65.44	23.34	6.45	0.31	3.34	0.79	0	0	0	0.34	0
11	70.14	20.99	5.13	0.19	3.21	0.34	0	0	0	0	0
12	70.06	22.16	4.67	0.23	2.84	0	0.04	0	0	0	0
13	72.15	20.99	3.82	0.05	2.99	0	0	0	0	0	0
14	65.47	20.97	8.23	0.34	4.98	0	0	0	0	0	0
15	73.76	18.23	4.08	0.11	3.64	0	0.18	0	0	0	0
16	75.01	20.42	2.54	0.06	1.91	0	0.07	0	0	0	0
17	70.47	20.17	5.15	0.19	4.02	0	0	0	0	0	0
18	73.79	22.66	2.11	0.05	1.39	0	0	0	0	0	0
19	70.19	23.72	4.09	0.04	1.95	0	0.01	0	0	0	0

The presence of carbides can also be seen an analysis of the cross-sectional area, Fig. 5-6. As can be seen in Fig. 5-6 a, there are thin lines, appearing bright in the SEM-photo, reflecting the solidification and microsegregation microstructure. They are decorated by numerous bright dots presenting carbides as can be seen from the close up given Fig. 5-6 b, c and the chemical EDX-analysis at the points 4 and 6. The carbides are again characterized by an increased Ti-content. Apart from carbides in Fig. 5-6 b, c there is a high P-containing phase, points 1 and 3, which has, apart from P, a strongly increased Mn-content. The points 2 and 5 are on the rim to the bulk phase and are characterized by high Mn- and Si-content. Finally, points 7 and 8 are in the bulk showing different degrees of microsegregation.

Fig. 5-6: Macro- and microstructure on a cross-section of 15-0.78-as-cast.

Table 5-3: Chemical composition measured at the marked points in Fig. 5-6.

	Fe	Mn	Si	Al	P	Ti	C	Nb	O
1	44.05	41.42	7.28	0.36	6.82	0.07	0	-	0
2	60.01	28.56	9.39	1.94	0.10	0	0	-	0
3	44.63	40.25	7.50	0.80	6.77	0.04	0	-	0
4	15.25	7.58	1.25	0.38	0	16.80	58.35	0.38	0
5	60.30	28.87	8.96	1.85	0.03	0	0	-	0
6	59.38	27.82	6.94	1.98	1.67	2.20	0	-	0
7	69.64	22.12	5.67	2.52	0.04	0	0	-	0
8	77.55	15.02	3.70	3.58	0.14	0	0		0

Se combined sulfides/selenides are found in X80MnSiAl 15 2.5 2.5 alloys (alloy from Table 5-4), as can be seen in Fig. 5-7. Because there is up to 0.3% Se in electrolytical produced maganese. Point 1 is a sulfide/selenide, Point 2 and 3 are carbides characterized by an increased Ti- and Nb-content. But as can be seen from Fig. 5-7, a the density of these precipitates is relatively low.

Table 5-4 Compostion of steel X80MnSiAl 15 2.5 2.5 alloys

Fe	Al	Si	C	P	S	Cr	Mo	Ni
79.7	2.46	2.48	0.684	0.0086	0.0091	0.0723	0.0018	0.0279

Fig. 5-7: Macro- and microstructure on a cross-section of a X80MnSiAl 15 2.5 2.5 alloy.

Table 5-5 Chemical composition measured at the marked points in Fig. 5-7.

	Fe	Mn	Si	P	Al	Ti	N	Nb	Se	S
1	11.11	45.63	0.39	1.29	0.40	0.62	0	4.73	10.74	25.09
2	20.81	10.53	1.27	1.04	0.40	47.32	0	17.36	0.04	1.22
3	47.15	17.87	4.19	0.83	1.94	19.42	0	7.83	0	0.77

Summing up, it has to be anticipated, that different types of precipitation affect the hot ductility of the investigated types of steel. At lower C-content ferrite formation plays an important role. At higher C-contents in the range of a pure austenitic solidification with it high degree of microsegregation carbide/cementite formation is of importance. Probably, cementite is encapsulated by a ferrite rim yielding an internal surface of reduced yield strength. Carbide and cementite formation may be enhanced by pronounced Si microsegregation due to its activity interaction with C. At some points strongly P-enriched phases are found on cross-sections. But as they are not found on the crack surfaces they may be of less relevance as long the P-content is sufficiently low. There are also indications that finely dispersed MnS-formation affect the hot ductility. Larger sulfides and selenides, in the case of alloying with Se-containing electrolytical produced manganese, may be less relevant. Like larger phospides they are not found on the crack surfaces. As a consequence of these findings, P, S and Ti should be kept low in high Mn steel. Also a low Se-content should be aimed also under working environment aspects. After annealing microsegregation and as well size as density of precipitates is significatly reduced leading to a significantly improved ductility, Fig. 5-8.

An acceptable ductility, e.g. reflecting in a corresponding elongation for the maximal stress in the shear test, does not mean allways that the crack susceptibility is also acceptable because this is esspecially affected by material inhomogenities. Of course, ductility and crack susceptibilty are in correlation to each other and both are improved by annealing and, furthermore, by processing like hot rolling.

Fig. 5-8: Microstructure of a X80MnAlSi 15 2.5 2.5 alloy before and after annealing.

5.3 Shear stress and elongation at high temperature

5.3.1 Influence of carbon on shear stress and elongation

Strength and ductility are given as a function of carbon content for the different Mn-contents in Fig. 5-9 for the engineering stress and in Fig. 5-10 for the true stress. As a general trend both strength and ductility increase with increasing carbon content.

As Fig. 5-9 and Fig. 5-10 show, the samples after annealing have significantly better elongation than as cast. It should be pointed out that samples having cracks on the cylindrical surface, nevertheless, could have good elongation, such as the samples 21-0.80, 17-0.65 etc.

As for 13Mn as cast samples, with carbon increasing, the true stress is increasing correspondingly. The true stress of the last sample 13-0.68 is low, because it has large cracks during the shear test. As for the 13Mn annealed samples, with carbon increasing, the true stress is increasing. In 15Mn and 17Mn, the same trend for true

stress was found. In 21Mn, the true stress values are scattering, because with exception of the first sample 21-0.10, the other samples all have cracks.

With Mn and C content increasing, the ferrite in high Mn microstructure is decreasing. Therefore, ferrite may be an important factor on the high temperature stress and elongation. The influence of ferrite is discussed in detail in the next section.

Fig. 5-9: Influence of C and Mn on engineering shear stress and elongation at 1000 ℃.

Fig. 5-10: Influence of C and Mn on true shear stress and elongation at 1000 ℃.

5.3.2 **Influence of ferrite on shear stress and elongation**

Carbon is an austenite forming element, which can make ferrite microstructure decrease. Ferrite contents achieved by ferriscope measurement of 13Mn and 15Mn samples are shown in Fig. 5-11. For higher Mn-contents no ferrite content could be measured, even so by a test with a strong Nd-Fe-B that there is still some ferrite. As cast samples have more ferrite than annealed ones, which means that the as cast state is far from equilibrium for 1020 °C which is approached by annealing. The ferrite transforms to austenite during the annealing process. Ferrite content in 13Mn as cast samples decreases from 56.7% to 0%, with carbon content increasing from 0.07% to 0.68%. Ferrite content in 13Mn annealed samples decreases from 42.56% to 0%, with carbon content increasing from 0.07% to 0.59%. Ferrite content in 15Mn as cast samples decreases from 17.26% to 0%, with carbon content increasing from 0.13% to 0.46%. Ferrite content in 15Mn annealed samples decreases from 1.98% to 0%, with carbon content increasing from 0.13% to 0.38%. Mn has essentially the same effect on ferrite content like carbon.

Fig. 5-11: Ferrite content in 13Mn and 15Mn samples

It can be seen from Fig. 5-12 using the engineering stress and Fig. 5-13 based on the true stress that, for the steel containing both ferrite and austenite, ferrite content is a very important factor on shear stress and hot ductility. With ferrite content increasing, the stress and ductility at high temperature tends to go down. But if ferrite content is

higher than 20%, the elongation is getting better for the 13Mn steel and probably also for the 15Mn grades. At room temperature, as for Fe-Mn-Al-C Triplex steel, ferrite has a positive effect on strength when its content is lower than 5%, and a higher content of ferrite (more than 30%) has a deleterious effect on mechanical properties [92].

Fig. 5-12: Correlation of engineering stress and elongation with ferrite content.

Fig. 5-13: Correlation of true stress and elongation with ferrite content.

Fig. 5-14: Schematic strength and ductility curves for limiting conditions.

The effect of ferrite on strength and ductility can be easily understood by considering the two limiting cases where the phases are in a serial and parallel morphology and making the assumption that there is a defined yield strength which is lower for the ferrite, Fig. 5-14. Of course, the real morphology is somewhere inbetween and there is no distinct yield stress at high temperatures, therefore, the real curves are between those for the limiting cases. The meaning of each symbol in Fig. 5-14 is shown as follows.

$$g_\delta = ferrite\ volume\ fraction$$

$$g_\gamma = ferrite\ volume\ fraction$$

$$\varepsilon = elongation$$

$$\varepsilon_\gamma = austenite\ elongation\ limit$$

$$\varepsilon_\delta = ferrite\ elongation\ limit$$

$$\sigma = \max shear\ stress$$

$$\sigma_\gamma = austenite\ stress\ limit$$

$$\sigma_\delta = ferrite\ stress\ limit$$

a)

$$\sigma = \begin{cases} \sigma_\gamma & g_\delta = 0 \\ \sigma_\delta & g_\delta > 0 \end{cases}$$

$$\varepsilon = \begin{cases} \varepsilon_\gamma & g_\delta = 0 \\ g_\delta \cdot \varepsilon_\delta & g_\delta > 0 \end{cases}$$

b)

$$\sigma = g_\delta \cdot \sigma_\gamma + g_\gamma \cdot \sigma_\delta$$

$$\varepsilon = \begin{cases} \varepsilon_\gamma & g_\delta < 1 \\ \varepsilon_\delta & g_\delta = 0 \end{cases}$$

5.4 **Three types of solidification in high Mn steel**

With carbon content increasing, three types of phase transformations were found in high Mn steel during solidification. Fig. 5-15 gives typical microstructures and line scans of three types of solidifications.

1. **Primarily ferritic transformation**

   Low carbon content: $L \rightarrow L + \delta \rightarrow \delta \rightarrow \delta + \gamma (\rightarrow \gamma)$

   Very low segregation during solidification ($l \rightarrow \delta$ transformation)

   Segregation due to $\delta \rightarrow \gamma$ transformation

   Fair hot ductility without cracks

2. **Primarily austenitic transformation**

   Middle carbon content: $L \rightarrow L + \delta \rightarrow L + \delta + \gamma \rightarrow \delta + \gamma (\rightarrow \gamma)$

   Low segregation during solidification ($l \rightarrow \delta, l \rightarrow \gamma$ transformation)

   Segregation due to $\delta \rightarrow \gamma$ transformation

   Good hot ductility without cracks

3. **Full austenitic transformation**

   High carbon content: $L \rightarrow L + \gamma \rightarrow \gamma$

   High segregation during solidification ($l \rightarrow \gamma$ transformation)

   Poor hot ductility with cracks

Fig. 5-15 Line scans of three solidification structures with low, middle and high carbon content

Fig. 5-16 was made to show high Mn phase transformation and the elements distribution in the microstructure. The main difference among the three types of transformations is that the origin of the δ phase:

Type 1: γ phase forms from δ phase;

Type 2: γ phase forms from both δ and liquid phase;

Type 3: γ phase forms from liquid phase.

Fig. 5-16: Three types of phase transformation.

The line scanning results are very interesting, so a line scan was done for each sample. Typical scans are shown in Fig. 5-16 and explained in Fig. 5-16. When the carbon is low, dark grey bcc ferrite is found, while full austenitic structure appears in high carbon sample. In the bcc zone, Al and Si are high, Mn is low, and in the full austenitic structure, linear zones of microsegregations are obvious. In this zone, Mn and Si contents are higher than in the matrix, and Al is lower. The reason is that the partition coefficients (concentration solid/concentration liquid) of Mn and Si are less than 1, while the partition coefficient of Al is more than 1.

Besides, from the shape of line scanning curves, there is a clear sharp boundary between ferrite and austenite structure, but the element distribution around segregation zone in the high carbon sample is gradual.

From the above analysis, it can be assumed that the phase diagram in Fig. 5-17 calculated by Thermo-Calc is wrong, because type 1 transformation cannot be found in it. The applied database in Thermo-Calc (2017a, Database TCFE8.1) software should be modified. But point A is a good indicator to distinguish between type 2 and 3 transformation, which is important for hot ductility analysis. If high Mn steel has more carbon content than point A, it probably has cracks during continuous casting. Therefore Al is of particular interest, because it can not only decrease steel's density, but also increase the δ ferritic phase zone which is good for crack susceptibility.

Fig. 5-17: Phase diagram of high Mn from Thermo-Calc (13%Mn 2.5%Al 2.5%Si).

5.5 **Microsegregation**

5.5.1 **Microsegregation ratio**

All the microsegregation ratio results are summarized in Fig. 5-18. According to the last section there are two types of microsegregation in this type of steel. The first is due to $l \rightarrow \delta / \gamma$ transformation and is characterized by positive Mn- and Si-segregation and by a negative Al-segregation. The corresponding max/min segregation ratios are given by the full symbols. The second is due to $\delta \rightarrow \gamma$ transformation and is characterized by a positive Al- and Si-segregation and a negative Mn-segregation. The corresponding max/min segregation ratios are given by the hollow symbols. This is because the partition coefficient k between liquid and austenite phase is less than one for Mn and Si, while it is more than one for Al and that between ferrite and austenite is less than 1 for Si and Al and larger than 1 for Mn. Their accurate value will be calculated in the next section. Even so the number of data is too small for a statistical sound conclusions there are some obvious trends. As long as there is a type 2 segregation, the solidification segregation (type 1) for Mn and Si is quite low. This is easy to understand as back diffusion is high as long as the solid phase is at least partly ferrite. The carbon range where type 2 segregation occurs decrease with increasing Mn-content. But even for 21%Mn there is a ferrite austenite transformation up to about 0.2%C. As long as ferrite is not removed completely by annealing, the segregation ratios in type 2 segregation are not significantly reduced by annealing. This is clear as these ratios are determined by the distributions coefficients between ferrite and austenite. Of course the ferrite mass fraction is reduced by annealing but this does not reflect in the segregation ratios. In contrast the segregation ratios during solidification segregation seem to decrease more or less graduately by annealing. In the as cast state the highest segregation ratios (min/max) are found for the lowest C-contents after disappearing of type 2 segregation. The reason may be the

effect of C on the distribution coefficient of Mn, Al and especially Si. So, the Si-activity coefficient in the melt is increased by C thereby increasing the distribution coefficient solid/liquid. The amount of microsegregation of Si due to solidification is after disappearing of type 2 segregation remarkable high. During type 2 microsegregation the enrichment of Al is dominant.

Fig. 5-18: Relationship of carbon and microsegregation ratio.

5.5.2 **Microsegregation modelling**

In this thesis, a microsegregation model was applied to determine k using experimental data, as described in section 3.4.2. From the line scan, the composition data of Mn, Al and Si in the microsegregation zone can be obtained, as Fig. 5-19 shows. There are three peaks in the figure for each element, so four groups of data can be used. The data in the front and end parts are both ignored, because they are not complete. The data between the peaks are equally divided into two parts. Partition coefficient e.g. of Mn yielding the best fit to the experimental data can be calculated. Fig. 5-20 shows that experimental data fits nicely with scheil equation curve for the resulting partition coefficient.

Fig. 5-19: Mn, Al and Si microsegregation in 13-0.68 as cast sample.

Fig. 5-20: Fitting of experimental data to scheil equation curve (Mn).

Table 5-6: Partition coefficient of Mn, Al, and Si

Group	Mn	Al	Si
1	0.87	1.24	0.49
2	0.77	-	0.69
3	0.85	-	0.35
4	0.85	1.18	0.68
Average	0.83	1.21	0.55

Al composition in groups 2 and 3 are rather chaotic, according to Fig. 5-19, so the data of these groups is ignored. As Table 5-6 shows, even though the peak values are different, the partition coefficients are almost the same, which is an advantage of this microsegregation model.

Fig. 5-21: Mn, Si and Al scheil solidification of sample 13-0.68 simulated by Thermo-Calc

Mn, Si and Al microsegregation in sample 13-0.68 was also simulated by Thermo-Calc software, as Fig. 5-21 shows. According to this, partition coefficients of Mn, Si and Al at the beginning of solidification are 0.70, 0.61 and 1.30, respectively. However, these values are not constant; as solidification continues, partition coefficients gradually tend to be 1. Thermocalc was also used to calculate the liquidus temperature for the investigated groups of high Mn-steels, Fig. 5-22.

Fig. 5-22: Liquidus temperature of high Mn steels calculated by Thermo-Calc (Al = Si =

2.5%).

6 Conclusions and outlook

Conclusions

1. High content of Mn and/or C make hot ductility of high Mn steels worse, because of the high microsegregation, coarse austenite columnar structure and formation of carbides. Si would make hot ductility even worse, because microsegregation ratio of Si is high.

2. After annealing, hot ductility becomes much better because of reduced microsegregation and dissolution of carbide precipitates.

3. High elongation at high temperature does not necessarily mean good hot ductility. Based on the experimental results, the samples with cracks can also have high elongation.

4. In the steel containing both ferrite and austenite, ferrite content is the main factor affecting high temperature stress and elongation. For the full austenitic steel, microsegregation ratio and precipitation may be the main factors.

5. Three types of solidification in high Mn steel are clarified: primarily ferritic transformation, primarily austenitic transformation and full austenitic transformation.

6. High Mn steel with high C contents can be produced by HSBC, because the strand undergoes no bending during casting. But the as cast strip should be annealed prior to rolling. In order to produce such fully austenitic steels on a conventional caster, a both ferritic and austenitic alloys ("Zone A") should be casted and subsequently annealed (1020 ℃, 1 hour) to remove its ferritic phase. Electromagnetic system is highly recommended to install in order to destroy the coarse columnar microstructure.

Outlook

In future investigations, the following aspects could be considered:

1. Influence of Si, Al, Cr, Ni, B and rare earth elements in high Mn steels on the high temperature shear stress and ductility, especially Al.

2. Influence of different temperatures and cooling rate on the shear stress and hot ductility, and comparison to the hot tensile test.

3. Reinforcement of the shear machine applied in this work to higher power, in order to test room temperature stress.

4. Influence of annealing temperature and duration on ferrite content, microsegregation, shear stress and ductility at high temperature.

7 **Appendix**

7.1 **Tensile test properties of different high Mn steels at room temperature**

Data from [39] [40] [41]

Number	Mn（wt. %）	Si（wt. %）	Al（wt. %）	C（wt. %）	Yield stress（MPa）	Ultimate tensile stress（MPa）	Uniform elongation（%）	Total elongation（%）
1	16.20	4	1.8	0.02	200	1100	28	39
2	17.90	3.20	2.90	0.02	460	930	39	46
3	15.90	1.90	3.50	0.01	430	580	38	47
4	18.20	4.30	1.80	0.06	260	1000	55	61
5	20.10	2.80	2.90	0.04	310	840	71	81
6	18.10	1.80	3.50	0.03	255	750	50	60
7	25.50	3.90	1.80	0.03	290	740	58	69
8	26.50	3.00	2.80	0.03	275	655	80	94
9	25.60	2.00	3.80	0.03	220	580	72	88
10	28.70	4.00	2.00	0.02	230	745	64	75
11	29.20	3.00	2.80	0.02	245	610	75	88
12	30.60	2.00	3.90	0.01	210	545	65	83
13	15.10	2.29	2.38	0.24	340	902	30	32

Table   continued

Number	Mn （wt. %）	Si （wt. %）	Al （wt. %）	C （wt. %）	Yield stress （MPa）	Ultimate tensile stress （MPa）	Uniform elongation （%）	Total elongation （%）
14	15.40	2.41	2.45	0.49	350	761	36	39
15	14.30	2.68	2.51	0.58	380	820	45	48
16	14.90	2.50	2.55	0.70	395	822	61	64
17	15.50	2.96	2.58	0.85	425	865	61	63
18	15.10	1.86	2.47	0.97	414	882	72	72
19	15.50	2.58	2.00	0.06	330	1100	30	32
20	15.00	2.50	1.50	0.05	300	1055	23	25
21	17.00	2.50	2.50	0.05	390	965	33	34
22	17.00	2.50	1.50	0.05	370	950	34	35
23	18.00	2.50	2.50	0.05	220	720	52	53

7.2 **Microstructures of high Mn steels**

13-0.07 as cast

13-0.07 annealed

13-0.17 as cast

13-0.17 annealed

13-0.26 as cast

13-0.26 annealed

13-0.30 as cast

13-0.30 annealed

13-0.43 as cast

13-0.43 annealed

13-0.51 as cast

13-0.51 annealed

13-0.59 as cast

13-0.59 annealed

13-0.68 as cast

13-0.68 annealed

15-0.13 as cast

15-0.13 annealed

15-0.20 as cast

15-0.20 annealed

15-0.30 as cast

15-0.30 annealed

15-0.38 as cast

15-0.46 as cast

15-0.46 annealed

15-0.69 annealed

15-0.78 as cast

15-0.78 annealed

17-0.1 as cast

17-0.1 annealed

17-0.17 as cast

17-0.2 annealed

17-0.35 as cast

17-0.35 annealed

17-0.44 as cast

17-0.44 annealed

17-0.51 annealed

17-0.58 as cast

17-0.58 annealed

17-0.72 annealed

21-0.10 as cast

21-0.16 as cast

21-0.16 annealed

21-0.22 as cast

21-0.22 annealed

21-0.32 as cast

21-0.32 annealed

21-0.43 as cast

21-0.43 annealed

21-0.55 as cast

21-0.55 annealed

21-72 as cast

21-0.80 as cast

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