1. Introduction

In the automotive industry, greenhouse gas emission reduction and improvement of vehicle safety are the main goals. New materials are considered to contribute significantly to this aim.\[1\]

In this respect steels combining both high strength and high ductility offer considerable benefits. Due to high strength, the sheet thickness can be reduced without loss of stability, thereby, a weight reduction is achieved. High ductility enlarges the degrees of freedom in the design of components. Furthermore, it yields high energy absorbing ability in a crash. A unique combination of high strength and ductility is provided by high manganese steel.

The properties of high manganese steels result from different mechanisms, such as crystallographic slip, the transformation of metastable austenite to martensite (TRIP),\[2\] twinning (TWIP),\[3,4\] and nano size κ-carbide formation (TRIPLEX).\[5\]

High Mn steel containing about 13 wt.-% Mn and up to 1.4 wt.-% C, first made by Sir Robert Abbott Hadfield in the 1880s, is famous for its high impact strength and resistance to abrasion.\[6\] By variation of the Mn-content from 5 to 30 wt.-% (medium to high Mn steels) and by alloying with Al, Si, and C the excellent mechanical properties of Mn steel can be further optimized and adjusted. The addition of Al and Si improves room-temperature properties by adjusting stacking fault energy and it also reduces density.\[7,8\]

Increasing C-content in a suitable range was found to increase both strength and ductility.\[9\] Microalloying with Ti, Nb, and V can be used to increase yield strength by precipitation hardening.\[10\] Thermomechanical treatment, e.g., by warm rolling at about 200 °C and subsequent annealing at 520–550 °C, adjusts deformation mechanisms and in this way the resulting properties.\[11\] Furthermore, properties can be adjusted by annealing, e.g., to control the carbide morphology.\[12\] Apart from the mechanical properties, various further aspects of high Mn steel properties have been investigated like crack formation mechanisms and the influence of hydrogen.\[13–16\] Hydrogen embrittlement and delayed fracture is a serious problem for certain high-manganese steels. Micro-segregation in high Mn steels can have a significant effect on the mechanical properties at ambient temperature.\[17–19\] Compared to fundamental aspects and the properties under application conditions, the behavior of high and medium Mn steels during production and processing has not found so much attention. The behavior during cold rolling has been investigated by Ofei et al.\[20\] The strain rate effect on the mechanical properties is important to adjust forming processes to the specific material and it is furthermore important for the crash performance in automotive applications.\[21\]

For casting,\[22–26\] hot rolling,\[27\] and welding,\[28,29\] the high-temperature properties are of crucial importance and it is essential to ensure a sufficient quality in these process steps. High-temperature brittleness restricts the applicability of the continuous casting process to certain groups of manganese
steels. The castability of medium manganese steels by thin slab casting has been assessed by Allam et al. Using a hot tensile test after in situ melting, various alloys were investigated to identify those which meet the ductility requirement of thin slab casting. The embrittlement of TRIP- and TWIP steel in the context of continuous casting conditions and the effect of different (micro) alloying and tramp elements have been analyzed by Mintz et al. based on a comprehensive literature review. Interaction with casting fluxes needed in continuous casting is of importance as well. Most of the problems arising during casting of high Mn steels can be avoided by strip casting as there is no bending of the solidifying product and no casting flux is required.

Mechanisms and fundamental aspects of embrittlement for high manganese steels have been investigated by several authors. Cabanas et al. have investigated by torsion tests the influence of the Mn-content on the hot deformation properties of austenitic binary Fe–Mn alloys containing 1–20 wt.-% Mn. An increase in the Mn-content was found to increase both the peak stress and the corresponding peak strain. For Hadfield-type steels, Chojeckia et al. have investigated the high-temperature embrittlement. Samples were cast and tensile tested after reheating. Micro-porosity and carbide formation were found to cause crack formation, where carbides could be removed to a great extent by heat treatment. High-temperature properties of three high-manganese steels with Mn- and C-contents between 9 to 23 wt.-% and 0.6–0.9 wt.-%, respectively, have been investigated by in situ melting hot tensile tests and hot compression tests by Bleck et al. Hot ductility, fracture characteristics, and flow during hot deformation were determined. Interdendritic fracture is reported to play an important role in crack formation. The influence of temperature, cooling rate, and strain rate on the high-temperature ductility of alloys with 17 wt.-% Mn and 0–9 wt.-% Al has been investigated by Borman et al. Depending on governing mechanisms like segregation and precipitation there may be different trends for the parameter on their effect on ductility. The high-temperature tensile properties of the three as-cast high-manganese steels with 21 wt.-% Mn, 5–6 wt.-% Al and 0.028, 0.28, and 0.64 wt.-% C have been investigated by hot tensile and hot compression tests at different temperatures and strain rates by Shen et al. It was found that steels with a carbon content of 0.028 and 0.28 wt.% have similar hot brittle temperature ranges, whereas the ductility of the steel improves with increasing temperatures for 0.64 wt.-% C.

The hot ductility of TWIP steels with 18–22 wt.-% Mn, 0.6 wt.-% C, 0.005–0.023 wt.-% N and with Al additions either low (<0.05 wt.-%) or high (1.5 wt.-%) have been examined by Kang et al. Poor ductility was found due to extensive precipitation of AlN at the austenite grain boundaries. It is stressed that sulfur should be low to avoid MnS formation. Hamada et al. have studied the hot ductility behavior of four high-Mn TWIP steels by hot tensile testing in the temperature range of 700 to 1300 °C at a strain rate of 1 s⁻¹. For high Al- and Si-contents (3 wt.-% each), the formation of the ferrite phase at high temperatures along the austenite grain boundaries was found to have the most deleterious effect on hot ductility. The effect of ferrite formation on the ductility in the temperature range around 600 °C was also reported by Yang et al. found at high-temperature tensile tests for steel with 15 wt.-% Mn, 0.6 wt.-% C, and 2.3 wt.-% Al. Furthermore, different types of precipitations like oxides, nitrides, sulfides, and selenides were found to contribute to high-temperature embrittlement.

Especially, considering the material behavior during the casting process the effect of micro-segregation on the high-temperature properties is of importance. Its effect often cannot be adequately assessed for material reheated from the room- to test temperature due to the resulting homogenization and recrystallization during cooling and reheating. Therefore, high-temperature tensile testing after in situ melting is preferably applied. This aspect is also taken into account by measuring the strength of a solidifying shell. Compared to low alloyed steels for high Mn steels, a significantly higher strength of the simulated solidified strand shell and an increased susceptibility for internal crack formation were found.

In our work, the mechanical properties of high Mn steels with 13 to 21 wt.-% Mn, 2.5 wt.-% Al and Si each and 0.05 to 0.8 wt.-% C are investigated by shear tests using a homemade apparatus enabling measurements in as-cast state directly after solidification. To assess the effect of the as-cast state and especially that of micro-segregation, the same material was tested again after reheating from room to test temperature and annealing there for one hour. To elucidate the effect of micro-segregation on strength and ductility its structure was analyzed by scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX) measurements. The results presented in this work are based on the doctoral dissertation of the first author.

2. Experimental Section

2.1. Melting

Experiments were performed on four series of steels with 13, 15, 17, and 21±0.3 wt.-% Mn. The aimed Al- and Si-content were kept constant at 2.5 ± 0.3 wt.-% each. The C-content was varied from 0.05 wt.-% to 0.8 wt.-% for each series. P and S were each <0.015, Ni <0.03, Cr <0.05 and Ti <0.02 wt.-%. Chemical analysis was performed by combustion analysis for C and S and by inductively coupled plasma atomic emission spectroscopy (ICP-AES). The nomenclature for the alloys used in this work is "wt.-% Mn–wt.-% C", e.g., "13-0.68" denotes steel with 13 ± 0.3 wt.-% Mn, 0.68 wt.-% C, 2.5 ± 0.3 wt.-% Si and 2.5 ± 0.3 wt.-% Al. The corresponding melt of ≈11 kg was made in a vacuum induction furnace using an alumina crucible. As base charge low alloyed steel was used. Mn was added as electrolyte manganese, Al and Si as pure metallurgical grade metals, and C as pure graphite. The melting and sampling were performed in an atmosphere of argon (>99.996 vol.% Ar) at approx. ambient pressure (950 mbar abs.).

2.2. Mechanical Shear Test

The steel strength and ductility at high temperatures were mostly determined by hot tensile tests after reheating the sample from the room- to test temperature or in situ after remelting prior to cooling to test temperature. Furthermore, torsion, compression, and bending tests are applied. To account for specific process conditions in continuous casting also special test equipment has been developed. In our work, a homemade shear test
apparatus was developed by the authors at the Clausthal University of Technology. Figure 1. The melt was sucked into a quartz tube submerged into the melt prepared inside the vacuum induction furnace. The quartz tube, which is optionally wrapped into an insulating ceramic felt to reduce radial heat loss and radial temperature gradients, Figure 1b, was fixed to a water-cooled sample holder made from copper. This sample holder was equipped with a type-B thermocouple in a quartz protection tube for continuous temperature measurement inside the solidifying cylindrical sample. The sample holder was screwed to a stainless-steel tube used to guide the cooling water and is connected via an inner second tube to the vacuum pump for melt sucking. This inner tube is also containing the thermocouple wires. To ensure a reliable connection between the sample and the sample holder, an anchor pin is fixed to the sample holder. After ∼2 min dwell time allowing for solidification, the complete equipment is removed through a vacuum lock from the furnace. Then the quartz tube was removed by crushing and the solidified steel sample inside is inserted into the hole of the shear test machine, Figure 1c,d. Solidification time is about 120–150 s and the cooling rate after the end of solidification is about 6 K s⁻¹. When the test temperature is reached, the shear was closed. The period between manual removal of the ceramic felt and the outer quartz tube and the start of shearing is about 10 s. This period should be short to minimize radial temperature gradients. During the test, the position of the moving shear plane (path length l) is measured by a transducer, the pressure P of the hydraulic piston is measured with an electronic sensor, and the center temperature of the sample close to the shear plane is measured by the above-mentioned thermocouple. From the geometrical data of the apparatus as well as from the pressure and the path data of the shear, engineering stress σ and elongation ε are calculated by

\[ \sigma = \frac{F(P,l)}{A_0} \]  

(1)

and

\[ \varepsilon = \frac{100(A_0 - A(l))}{A_0} \]  

(2)

whereby \(A_0\), \(A(l)\), and \(F(P,l)\) are the cross-section area of the sample, the overlap area of the holes in the fixed and the moving plates of the shear, and the force acting on the shear plate, respectively. \(A(l)\) can be easily calculated as a function of path length \(l\) based on basic geometrical considerations. \(F(P,l)\) is obtained as a function of the hydraulic pressure \(P\) and of the path length \(l\) which determines the lever arm configuration. Alternatively, a true stress can be calculated by replacing \(A_0\) in Equation (1) by \(A(l)\). In principle \(\varepsilon\) calculated by (2) can alternatively be considered as a reduction of area number.

This type of shear test, even though the setup used is still provisional, offers the following benefits: 1) The diameter of the cylindrical sample being 23 mm provided a representative average of the material’s properties. Occasional smaller porosity had no significant influence. Thus, in that shear test, a good reproducibility was found; 2) No complicated sample preparation is required, so the time needed per test is quite short; 3) The composition of the steel melt can be adjusted after each sampling. In this way, a test series with increasing contents of certain elements can be performed in an efficient way; 4) Due to the optional thermal insulation of the outer cylinder surface by a fiber felt, the heat flux had a larger axial component and, therefore radial temperature gradients in the sheared area can be minimized; 5) The cooling rate can be controlled within certain limits by the distance of the shear plane from the water-cooled sample holder and optionally by placing an insulating layer between steel and sample holder. In a more advanced setup than that used in this work, inductive heating could be used to reduce the cooling rate, e.g., to simulate continuous casting conditions characterized by temperature cycling; 6) The thermocouple was placed a few millimeters away from the shear plane so that there is good control of test temperature; and 7) During the shear test usually internal cracks were generated in the material close to the shear plane. As these internal cracks, in contrast to the shear surface itself, are protected against oxidation, the structure of the crack surfaces is perfectly preserved and is especially suitable for further investigation.
A disadvantage of this test setup is that its results are not independent of the geometrical parameters of the shear. They depend, e.g., on the sample diameter and the radius of the cutting edge which is 1 mm in this work, Figure 1a. Actually, real cutting should be avoided to achieve shearing. Therefore, the results must be considered semi-quantitative and can be used so far only for comparison which each other.

To assess the effect of the initial solidification structure and micro-segregation on the mechanical high-temperature properties, the second series of shear tests were performed with the cut off lower part of the sample. In this test, the as-cast sample previously cooled to room temperature was annealed for 1 h in a muffle furnace in air. A K-type thermocouple inserted into a dummy cylinder of similar material and geometry placed adjacent to the test sample was used to control the temperature during annealing. The annealing temperature was chosen to be about 20 °C above the test temperature, considering the sample cooling rate during the transfer of the sample from the muffle furnace to the shear test.

2.3. Metallography and Structure Analysis

The shear test samples were cut a few millimeters away from the shear surface, grinded, and polished. The solidification and grain structure were analyzed by SEM. Special attention was paid to internal cracks developing during the shear test. The micro-segregation was determined by line scans using EDX and is expressed by the micro-segregation ratio. This is obtained for the corresponding element by dividing the local concentration by the average concentration over all points of the line scan. The average ferrite content (in vol.-%) was measured by a commercial ferrite scope (FERITSCOPE FMP30). The principle of this device is based on the magnetic induction method. A magnetic field generated by a coil interacts with the magnetic fractions of the specimen. The changes of the magnetic field induce in a second coil a voltage proportional to the ferrite content.[41]

3. Results

All shear test results given in this article are for a test temperature of 1000 °C, which is a typical temperature range, e.g., for bending and straightening in a continuous casting process or for in-line hot rolling in strip casting processes. Annealing was for 1 h at a temperature of 1020 °C. The reason for choosing 1020 °C was that considering temperature loss during transfer of the sample from the furnace to the test apparatus, a test temperature of close to 1000 °C is obtained. This allows a direct comparison of the data for the as-cast and the annealed state to identify the effect of micro-segregation diminished by annealing. This is a major aspect of this work, the objective is not to identify favorable annealing temperatures. The solidification time was in the range of 120–140 s. The cooling rate after solidification was 5–6 °C s⁻¹. The elongation rate was 40% s⁻¹ for all shear tests.

3.1. Mechanical Properties

The high-temperature behavior in the shear test differs considerably between the as-cast and the annealed state as can already be seen from the shear surfaces, Figure 2. In the as-cast state, a scaly surface structure is observed reflecting a stick and slip behavior during shearing. On the outer cylinder, surface cracks appear with an increasing tendency with increasing C-content. In the annealed state, the shear surfaces are much smoother and there are much fewer surface cracks on the outer sample surface. The described behavior and the crack surface appearance given in Figure 2 for 13–x is typical also for 15–x, 17–x, and 21–x.

Figure 3 shows for example the stress-strain curves for 13–x and 21–x both recorded for the shear tests in the as-cast and annealed states. From such curves, σmax and εmax are obtained as the highest value of σ and the corresponding elongation ε. There are again obvious differences between the as-cast and the annealed state. In the as-cast state, especially for 21–x, after reaching σmax the strength does not drop linearly like for the annealed state. It can be assumed that this behavior results from crack formation in the sample, maybe internal cracks in the shear plan or cracks on the outer sample surface. The crack formation results in a stick-slip behavior during the shear test. For 13–x, there is a clear effect of the C-content. At constant elongation of, e.g., 20%, the strength is increasing with increasing C-content up to about 0.4 wt.-% C. Beyond that level the strength at 20% elongation stays constant. In contrast, for 21–x there is no clear effect of the C-content on the strength up to an elongation of about 40%.

Figure 4 shows εmax and σmax as a function of the C-content for 13–x, 15–x, 17–x, and 21–x both in the as-cast (dashed lines) and in the annealed state (solid lines). For 13–x and 15–x, the ferrite volume content measured at room temperature is given as well. With the available ferrite scope, no reliable measurements were possible for 17–x and 21–x. For most of the investigated alloys ductility is clearly improved by annealing. For 13–x, both stress and ductility strongly increase with increasing C-content up to about 0.5 wt.-%. This increase correlates quite well with the decrease of the ferrite content which is known to result in embrittlement.[36] At higher carbon contents in the as-cast state strength and ductility decrease again strongly. This is mainly due to primary carbide formation as discussed in more detail in section 3.3. After dissolving these carbides by annealing strength and ductility are significantly improved. For 15–x in the as-cast state, reduced strength and ductility are found for lower (ferrite embrittlement) and for high C-contents (embrittlement by carbides). In the annealed state containing a few ferrites, there is an increase in strength and ductility with increasing C-content. The effect of annealing is again especially pronounced for the highest carbon content. The alloy group 17–x also exhibits a slight increase in strength and ductility with increasing C-content. Even though it could not be measured with the ferrite scope, it can be assumed that there is still some ferrite present in the as-cast state at lower C-contents. Thus, there is a clear effect on ductility by its removal after annealing. It is remarkable that 17–x is the only of the four investigated alloy groups, where annealing reduces strength within the complete C-range. For 21–x, the strength is relatively constant in the low and medium C-range. There is some scattering of the strength data but the difference between the as-cast and the annealed state is quite small. Assuming this scattering is due to variation in the alloy composition. At higher carbon contents, susceptible to carbide formation, the effect of annealing becomes significantly larger.
Figure 5 shows for the alloys with 13 and 15 wt.% Mn the maximum strength and ductility as a function of room temperature volume fraction of ferrite, both for the as-cast and the annealed state. To avoid interference with carbide formation, only alloys with C < 0.6 wt.% were considered. As a function of ferrite content, there is no distinct difference between the as-cast and annealed state. Despite a certain scattering, there is a clear trend of decreasing strength with increasing ferrite content. The same applies to ductility up to a ferrite content of /C25/20 vol.-%. Thereafter, the ductility stays constant or even increases a little bit again. Depending on the morphology, a two-phase structure with different yield strengths of the phases has a lower ductility than each of the single phase with a minimum at a certain intermediate phase fraction. Having the role of ferrite fraction in mind, the reason that 13–x at low C-contents show no significant effect of annealing (Figure 4) may be explained by the fact that annealing reduces the initially high ferrite content only to an extent that does not influence strength and ductility significantly. On the other side, it is reasonable that annealing has no significant effect on 21–x at lower C-contents (Figure 4) as there is practically no ferrite in both states. It should be mentioned that according to results obtained, e.g., by Thermo-Calc even for 13-0.1 and 15-0.1 there should be no ferrite at 1000 °C in equilibrium. Thus, micro-segregation obviously plays a dominant role.

3.2. Micro-Segregation

For the type of alloys under consideration with high contents of both austenite (Mn, C) and ferrite (Al, Si) stabilizing elements, special conditions for micro-segregation are found. Figure 6 shows mappings of Mn, Al and Si for 15-0.06 as cast. Two types

![Figure 6](image-url)
of segregation can be recognized. The larger structure (type 1) characterized by high Mn- and low Al-concentrations results from phase transformation from liquid to solid, and the finer one (type 2) results from ferrite to austenite transformation. This type 2 of micro-segregation is characterized by high Al- and low Mn-contents.

Figure 3. Stress–strain curves for 13–x and 21–x both in the as-cast and annealed states. There is an obvious difference between the as-cast and annealed states. In as-cast state, after reaching the maximum the strength does not drop linearly due to crack formation in the sample resulting in stick-slip behavior. For 13–x, there is a clear effect of the C-content. In contrast, for 21–x this effect is smaller below 40% elongation.

Figure 4. Ductility ε_{max}, strength σ_{max}, and ferrite volume content as a function of the C-content. Strength and ductility tend to increase with C-content and for 13–x and 15–x clearly with decreasing ferrite content. Ferrite content is reduced by annealing and ductility is increased by annealing. The effect of annealing becomes larger for the higher C-contents (C > 0.6 wt.%).
In type 2 segregation, the austenite grows into the initially formed ferrite. In this way, the ferrite is enriched with Al and Si and the austenite with Mn and C. As a result, the ferrite is increasingly stabilized until the transformation stops.

Therefore, ferrite can be present at high testing temperatures although the alloy would be fully austenitic in equilibrium. The segregation ratio of Mn, Al, and Si obtained by EDX-measurement along a line scan for 13-0.30 and 21-0.32 each as-cast is shown in Figure 7. For a point on the line scan, the segregation ratio of the corresponding element is calculated by the content at the specific point divided by the mean of the contents at all points. The different characteristics of the two types of segregation can be well distinguished in Figure 7. When the solidification takes place mainly as liquid to austenite transformation, quite high segregation ratios for type 1 segregation are found for Si.

From line scans like that in Figure 7, the maximum segregation ratios are obtained. They are defined for each element as the highest (for enriched elements) or lowest (for depleted elements) segregation ratio measured on the line scan. These maximum segregation ratios of Mn, Al, and Si for the alloys 13–x, 15–x, 17–x, and 21–x are given in Figure 8 for both types of segregation. The range of C-contents at which type 2 segregation occurs (hollow symbols in Figure 8) shrinks with increasing content of the austenite stabilizing elements Mn and C. It is further decreased by annealing. For 17–x and 21–x, there is type 2 segregation up to about 0.2 wt.-% C in the as-cast state. After annealing in 21–x, no type 2 segregation is found anymore. The C-areas where type 2 segregation occurs correspond quite well with those where larger ferrite fractions were found for 13–x and 15–x (Figure 4 vs 8). It is noteworthy that the maximum segregation ratios for type 2 after annealing are approximately the same as in the as-cast state, whereas the amount of ferrite is significantly reduced by annealing as can be seen from Figure 4. This can be explained by a quasi-heterogeneous equilibrium between ferrite and austenite. So, during annealing, the amount of ferrite is reduced, whereas the concentrations in both phases are only slightly changed. Thus, having in mind the correlation between ferrite content and strength and ductility (Figure 4), it can be concluded that embrittlement in the lower C-area is mainly caused by type 2 segregation and the associated ferrite formation. The level of type 1 segregation is low for smaller C-contents (Figure 8) because when a larger fraction of Mn, Al, and Si for 15-0.06 as cast. Brighter color indicates higher concentrations. Two types of segregation can be recognized. The larger structure (type 1: high Mn and low Al) results from liquid to solid transformation, and the finer one (type 2: low Mn and high Al) results from ferrite to austenite transformation. Si is enriched by both types of solidification but type 1 segregation for Si is low due to mainly ferritic solidification and back diffusion.
Figure 7. Segregation ratio obtained by EDX-measurement along a line scan for 13-0.3 and 21-0.32 as cast. Different types of segregation are found. The two types of segregation can be recognized for 13-0.30, left diagram. Type 1 (high Mn, low Al) results from solidification, and type 2 (low Mn, high Al) from ferrite to austenite transformation. Type 2 segregation preserves the ferrite down to low temperature. High Si segregation is found for completely austenitic solidification, right diagram for 21-0.32.

Figure 8. Maximum segregation ratios of Mn, Al, and Si for both types of segregation as well in as-cast as in the annealed state. Type 2 segregation is favored by a lower level of austenite stabilizers (Mn, C) and at the same time type 1 segregation is reduced by back diffusion in the ferrite. Mainly austenitic solidification leads to high Si segregation which has its highest level close to the point where type 2 segregation disappears. Afterward, Si segregation decreases with increasing C-content. Annealing reduces the segregation level for most elements.
of solidification takes place from liquid to ferrite, back diffusion into the solid is larger due to higher mobilities in ferrite compared to austenite. The highest segregation levels for type 1 are found for Si and in the as-cast state close to the end of the C-area where type 2 segregation exists. With increasing C-content, the level of type 1 segregation for Si in the as-cast state decreases, which may be because of the effect of C on the distribution coefficient of Si between austenite and liquid. Annealing reduces the segregation level especially for Si and Mn for type 1 segregation, whereas its effect on Al is relatively weak.

The embrittlement for carbon contents larger than ≈0.6 wt.-% C and the significant improvement by annealing (Figure 4) can be related to type 1 segregation. In the segregated zone of final solidification where Mn, C, Si and tramp elements like Ti, Nb, P, S, and Se (if electrolytic Mn is used) are enriched, different types of precipitates like oxides, carbides, silicides, sulfides, selenides, and phosphides may form.\[42-45\] Figure 9 shows on the left side a SEM figure of 15-0.69 as cast and on the right the same alloy after annealing. The segregated zones in the as-cast state are visible as bright lines. Assumingly these zones are ferritic at room temperature, maybe due to the enrichment of Si and by the C depletion in the matrix due to carbide formation. By annealing, the ferrite is removed but the corresponding zones are still visible as darker areas in the SEM photo. After disappearing of the ferrite, other types of precipitates like sulfides/selenides phosphides, etc. formed in the segregation zone, become visible.

### 3.3. Crack Formation

The embrittlement is associated with internal and external cracking. Figure 10 shows cracks formed close to the shear plane for 15-0.78 as cast on a polished cross-section.

The area marked in the left figure is magnified in the two adjacent figures. The two figures on the left were obtained by backscattered-electron imaging making the cracks well visible. In the right figure obtained by secondary electrons-imaging, the micro-segregation pattern can be recognized more clearly. Cracks are predominately found at the interface of domains with different solidification orientations. These domains for those alloys are mostly coincident with the austenite grains.\[46\]

It can be assumed that segregation is especially high in these locations. Figure 11a–e shows SEM-photos of cracks for 13-0.68 as cast. The positions of the presented sections are marked by squares in those figures with lower magnification. In some cases, there is a dimple-type fracture with small craters on the crack surface, Figure 11b,c. This type of fracture often results from void formation starting at precipitates. Various types of precipitates are reported to be present in the type of steel under consideration like Al2O3, AlN, MnS(Se), and also complex ones like AlN–MnS, AlON–MnS, and AlON.\[42-44\] In some areas, dendritic structures become visible, Figure 11e, with a dendrite arm spacing in the order of magnitude of 1 μm. At some of the marked points in the figures depicting the dendritic structure, high Ti-contents as large as 12.7 wt.-% and Nb-contents up to 2 wt.-% were measured by EDX, whereas the Ti- and Nb-content in the base material is 0.01 to 0.02 wt.-%. Such a morphology and an enrichment of carbide forming elements especially in the trunk of the dendrites is typical for primary carbide formed in the liquid close to the end of solidification.\[45\] The main components always measured at the marked points are Fe and Mn. Therefore, it is quite likely that these dendritic structures are carbides formed in the center zone of type 1 segregation. Figure 11f shows an internal crack for 15-0.78 as cast. The concentration on the crack surface (positions (3) and (4)) corresponds to that found for type 1 segregation on the line scans. In some cases, the structure below becomes visible (position (5)). The Ti-content of about 10 wt.-% and the Nb-content of about 2 wt.-% measured at position (5) leads again to the conclusion that this is also a carbide dendritic structure. Thus, it is quite likely that the observed embrittlement in the upper carbon range in the as-cast state is due to the inner interfaces formed by the primary carbides.
or due to a local decarburization in the vicinity of the carbides 
leading to a locally decrease of yield strength.

4. Summary and Conclusions

The mechanical high-temperature properties of steels with 13 to 21 wt.-% Mn, about 2.5 wt.-% Al and Si, respectively, and 0.07 to 0.8 wt.-% C were investigated at 1000 °C both in the as-cast and annealed states. The as-cast material is tested directly after solidification and cooling to 1000 °C, whereas the annealed material is tested after reheating the material from room temperature to 1020 °C and soaking for one hour. A homemade shear test apparatus was used to measure strength and ductility. The reproducibility of the test results was found to be good for such types of measurements. However, it must be emphasized that the obtained data depend to a certain extent on the geometrical parameters of the test apparatus. Therefore, the data achieved so far can only be used for comparison among themselves.

For the mechanical properties, there is a trend of increasing strength and ductility with increasing C-content, whereas there is no clear trend for the Mn-content. Annealing mostly improves strength and ductility. The mechanical properties are significantly influenced by micro-segregation. Two types of micro-segregation are found for the investigated high manganese steels. Type 1 is the usual one related to solidification and is characterized by an enrichment of Mn and Si whereas the Al-content is reduced. Type 2 results from ferrite to austenite transformation for alloys with complete or partial solidification from liquid to ferrite. It leads to an enrichment of Al and Si and to a reduction of the Mn-content. This type of micro-segregation is predominantly found for lower contents of the austenite stabilizer C and Mn. Type 2 segregation is even found for 21 wt.-% Mn up to 0.2 wt.-% C. Due to type 2 segregation, ferrite exists at high temperatures where only austenite would be present in equilibrium. Both strength and ductility are thereby affected. For C < 0.6 wt.-% and 13 and 15 wt.-% Mn, strength and ductility were found to correlate quite well with the ferrite content (measured at room temperature) regardless of whether the state is "as cast" or "annealed". Material properties are changed by annealing to that extent the ferrite content is reduced. Apart from annealing, the embrittlement by ferrite formation can only be reduced by avoiding critical compositions, e.g., by reducing the contents of ferrite stabilizing elements or increasing that stabilizing austenite. In the intermediate carbon range, type 2 micro-segregation disappears and solidification takes place mainly by liquid to austenite transformation. The resulting type 1 micro-segregation may lead to locally high Mn and especially Si-contents. Annealing reduces the high Si segregation significantly but this does not lead to a similar large change in strength and ductility. Despite the high Si-contents also the segregated zones might be austenitic at 1000 °C and, therefore, the material is homogeneous and more ductile. For C-contents larger than ≈0.6 wt.-% C the as-cast strength and ductility are again significantly below those after annealing. By investigating internal cracks formed close to the shear plane using SEM, precipitates, in particular dendritic carbide structures, were found as a possible reason for the embrittlement. High Ti- and Nb-contents are found in these carbides, therefore, it may beneficial for the high-temperature properties to keep the concentrations of carbide-forming elements low in such type of steels. Of course, these elements may be advantageous for the room temperature.
properties, especially for the yield strength.[10] Precipitates formed in the micro-segregation zones can be removed to a certain extent by annealing, whereby the amount of micro-segregation is reduced as well. Si by its effect on the C-activity may increase the driving force for carbide formation and, therefore, could be critical for the high-temperature properties at higher C-contents. Thus, reduction of Si-content should help to improve high-temperature ductility. In contrast, Si may be beneficial for the properties at room temperature under service conditions. Furthermore, Mn-sulfides/selenides and phosphides are found in the segregated zones, hence, P, S, and Se should be kept as low as possible.

Acknowledgements

The support of the China Scholarship Council is greatly appreciated. Open Access funding enabled and organized by Projekt DEAL.

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

as-cast properties, carbide precipitation, ferrite formation, high Mn steel, hot ductility, shear tests

Received: March 7, 2022
Revised: July 12, 2022
Published online: July 28, 2022

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