Novel nanostructured bainitic steel grades to answer the need for high-performance steel components (NANOBAIN)
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(NANOBAIN)

T. Sourmail, V. Smanio
Ascometal CREAS
Avenue de France, BP 70945, 57301 Hagondange Cedex, FRANCE

C. Ziegler, V. Heuer
ALD Vacuum Technology GmbH
Wilhelm Rohn Str. 35, Hanau, GERMANY

M. Kuntz
Robert Bosch GmbH
Materials and Processing Dept, P.O. Box 300240, Stuttgart, GERMANY

F. G. Caballero, C. Garcia-Mateo, J. Cornide
CENIM-CSIC
Dept of Physical Metallurgy, Madrid, SPAIN

R. Elvira
Gerdau I+D
S. A. Barrio Ugarte, 48971 Basauri, SPAIN

A. Leiro, E. Vuorinen
Lulea University of Technology
Lulea, SWEDEN

T. Teeri
Metso Minerals Oy
PO Box 237, 33101 Tampere, FINLAND

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1. INTRODUCTION: STATE OF THE ART AT PROJECT ONSET

The background presented in this section summarises the recent discovery of low-temperature bainitic steels (Nanobain steels) and their present basic mechanical properties. Because this is a very recent breakthrough, complex properties such as wear and fatigue resistance had not yet been investigated at the onset of the project. Published results nevertheless suggested that Nanobain steels should perform outstandingly in these areas.

1.1 THE DEVELOPMENT OF LOW-TEMPERATURE BAINITE

1.1.1 A microstructure with exceptional mechanical properties

For their good combination of strength and toughness, bainitic steels are increasingly replacing quenched and tempered steel grades, introducing cost savings that can alone make the difference between profit and loss in some cases. [HKDB, 2011a]. Considerable progress has been made since the early bainitic steels, which has lead to improvements in the performances of these steels. The addition of a high silicon content (~1.5 wt% and above), in particular, has allowed for the suppression of cementite, leading to a microstructure consisting of bainite and residual austenite. Such microstructures can exhibit excellent combinations of strength and toughness (1600-1700MPa, 130 MPa.m1/2), comparing, for instance, with the considerably more expensive high Ni maraging steels [FC, 2001].

![Image of nanostructured bainite](image)

Carbide free bainitic steels derive their strength essentially from their fine grain size, typically below 1 µm (the relevant dimension being the plate thickness). A process that would allow further grain refinement bears the promise of unattained combinations of strength and toughness in economical steel grades. For example, there is considerable work being devoted to obtaining nanostructured grain sizes in bulk steels by use of severe plastic deformation (SPD), with more than 1500 scientific publications published on this topic over the last ten years [TCL, 2003]. However, it is unlikely that any of the processes used for SPD can ever be applied for economical industrial production. In addition, the grain sizes resulting of process such as equal channel angular pressing (ECAP, one of the few SPD processes able to work on bulk material rather than surface only), are seldom less than a few hundred nanometers [KTP, 2002] and have very little resistance to tempering.

By contrast, recent advances in the understanding of the bainitic transformation can be exploited to generate nanocrystalline structures by a simple heat-treatment of carefully designed alloys, without the need for expensive processes or elements. With careful alloy design, bainite transformation can be made possible at temperatures as low as 125 C, and can be exploited to achieve strength in excess of 2.5 GPa with considerable toughness (30 MPa.m1/2) [FGC, 2004].
These microstructures have, in addition, an unexpectedly high tempering resistance, exhibiting a hardness still over 600 HV after 1 h at 550 °C. The discovery of these nanobainitic microstructures is potentially one of the most significant ones in steel metallurgy over the past 10 years, and it is these microstructures that the present proposal is concerned with.

1.1.2 The kinetics of low temperature bainite formation

In the initial development, low temperature bainite was found to exhibit exceptional mechanical properties, but considerable time (up to several days) was required to allow for completion of the bainitic transformation.

On the basis of thermodynamic understanding of the bainitic transformation, composition modifications have been successfully implemented to accelerate the bainitic transformation at low temperatures [CGM, 2003a].

1.2 Wear properties of bainite

1.2.1 Introduction

As the development of low-temperature bainite is extremely recent, complex mechanical properties such as fatigue, or system properties such as wear, have not yet been investigated, and no results are available at the industrial scale. Nevertheless, work carried out on conventional bainitic steels can provide useful information, and the following summarises briefly the work carried out on the wear of bainitic steels, most of which was concerned with rail steels.

1.2.2 Sliding/rolling wear resistance

Wear properties of bainitic structures have essentially been studied in the context of rail steels. Rail steels have long been manufactured using pearlitic steels, the wear resistance of which increases with their hardness [JCI, 1980]. As bainitic microstructures offer much higher hardnesses for lower carbon contents, much research has been devoted to replacing pearlitic steels with bainitic ones. This is because the latter were expected to offer improved wear resistance with better weldability.

The early work on wear resistance of bainitic steels for rails produced disappointing results both in the laboratory and on the tracks [JCI, 1992] and [JCI, 1996], with reported rolling/sliding wear rates up to ten times higher than those of pearlitic steels. As explained below however, these pessimistic early conclusions came from an oversimplified understanding of what constitutes bainite. As pointed out by Clayton and Jin [JCI, 1996], four types of bainitic microstructures should be distinguished: upper and lower bainite, carbide-free bainite and globular bainite. Not all bainites perform equally during wear tests.

The work of Clayton [JCI, 1980] and Clayton et al. [JCI, 1987] suggested that bainitic microstructures did not perform significantly better than pearlitic ones in dry rolling/sliding tests except at relatively low hardness. In further experiments on low carbon bainitic steels however, it was found that an 0.04 %C bainitic steel outperformed harder pearlitic steels in rolling-sliding wear tests [SDe, 1991]. Later, Clayton and Devanathan [JCI, 1992] investigated the wear of 50 HRC bainite formed by isothermal treatment at 290 °C and reported a far higher wear resistance than for a pearlitic microstructure. Interestingly, the same material transformed to a bainitic microstructure of hardness equal to that of the pearlitic one did not perform as well as the latter.
Figure 2: Wear rate of different bainitic steels as a function of hardness, after Clayton and Jin.

These results, in contrast with previous conclusions by other authors [JKa, 1985], suggested that much was to be gained from an increase in bainite hardness (for lower bainite at least). Additional results confirmed the excellent potential of bainitic microstructures, as Clayton and Jin [JCl, 1996 and 1987] reported exceptional wear resistance in high silicon cast bainitic steels (Fig. 2) and confirmed that carbide free structures gave the best wear resistance (rolling/sliding).

From their results, the authors clearly anticipated that increasing the hardness while retaining the carbide free microstructure should enable outstanding wear resistance. It should therefore be underlined that the design of low-temperature bainitic steels will result in progress in exactly that direction, and is therefore expected to lead to excellent wear resistance (dry sliding/rolling conditions).

Since then, there are numerous additional reports of the good sliding wear performance of carbide free bainitic steels, for example, Shipway et al [PHS, 1997], or Vuorinen et al [EVu, 2006], who reported excellent results on spring steels (0.6C, 1.9Si) isothermally transformed to bainite, both in dry sliding wear tests and in service (knives in a hammer mill used for cutting saw dust). All these progresses have led to the development of bainitic steels with excellent wear resistance and high toughness [HKDB, 2005].

The excellent results obtained with carbide free bainitic structures are generally attributed to the combination of high strength and toughness resulting from the ultrafine microstructure and absence of carbides.

1.2.3 Abrasive wear resistance:

There are significantly fewer data available on the abrasive wear resistance of bainitic structures (and in particular of carbide free bainitic structures). However, some results can be of interest: Liu et al [JLi, 1997], for example, have drawn a comparison between high Cr cast irons and austempered nodular cast irons. The austempered nodular cast irons have a bainitic matrix obtained by isothermal transformation. These exhibited abrasive wear resistance similar to that of the high Cr cast irons, at a manufacturing cost significantly lower [JLi, 1997]. A study of the mechanisms of abrasive wear suggested that optimum resistance should be obtained for a combination of high strength and toughness [AKJ, 2003].

1.3 Fatigue of bainitic microstructures

It is well established that, for an identical cleanliness level, the fatigue resistance of steels is mostly related to its UTS: the higher the UTS, the higher the fatigue resistance. In this regard, as Nanobain steels can exhibit very high UTS, they are expected to perform significantly better than conventional, lower strength steels.
In addition, the role of the carbide free microstructure is of particular interest. Whilst the majority of bainitic steels are constituted by a mixture of bainitic ferrite and carbides, high Si (or high Al) bainitic steels contain a mixture of bainitic ferrite and residual austenite. It had initially been thought that residual austenite would be detrimental to fatigue resistance, because its transformation in the vicinity of the fatigue crack tip results in an increase in strain hardening rate [HKDB,2001a]. However, experimental results have suggested the opposite, as illustrated in Figure 3.

![Figure 3: Crack growth rate vs stress intensity range for a high Si bainitic steel with different heat-treatments. The thresholds are not significantly different although the steel with higher residual austenite has a markedly lower UTS. After Wenyan et al, 1997.](image)

The results of Wenyan et al. [LWe,1997] indicate that residual austenite could have a strongly beneficial role on fatigue resistance. An explanation suggests that the strain hardening rate approach neglects the work required to transform the residual austenite, and therefore fails to predict the beneficial influence of the latter [HKDB,2001a]. As of today however, fatigue properties of ultrafine bainite ('Nanobain' steels) have not been investigated.

1.4 OVERVIEW OF WORLDWIDE PROGRESS DURING THE COURSE OF THE PROJECT

Activity has been sustained on the topic of nanoscaled bainitic microstructures (or 'super-bainite') over the duration of the project, and a number of results are now published concerning wear and fatigue of these microstructures [JAC,2011; MJP,2011; AST,2011; TSW,2008; MNY,2011; PZh,2011]. Whilst fatigue results published to date should be taken with caution, wear data seem to confirm interesting performances from these microstructures.

2. WORK CARRIED OUT AND MAIN RESULTS

2.1 WP1: DESIGN OF ALLOYS

The objective of WP1 was to design alloys amenable to bainitising at low temperatures, with sufficient hardenability for the quenching process to be used (gas quenching for the smaller components, salt-bath for the larger ones). This has to be balanced with the need to maintain reasonably fast transformation kinetics and thus lean compositions.

This has resulted in the design of 9 alloy compositions that were manufactured as laboratory casts (Table 1).
Table 1: Proposed chemical compositions of experimental casts.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>1C-Nb</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>0.03</td>
</tr>
<tr>
<td>1C-Mo</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>0.10</td>
<td>---</td>
</tr>
<tr>
<td>1C-2.5Si</td>
<td>1.00</td>
<td>2.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.8C</td>
<td>0.80</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C-2.5Cr</td>
<td>0.60</td>
<td>1.50</td>
<td>0.75</td>
<td>2.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C-Nb</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>---</td>
<td>0.03</td>
</tr>
<tr>
<td>0.6C-Mo</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>0.10</td>
<td>---</td>
</tr>
</tbody>
</table>

Table 2: Proposed chemical compositions for industrial heats.

<table>
<thead>
<tr>
<th>Reference</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CSI-indus</td>
<td>1.00</td>
<td>0.75</td>
<td>2.50</td>
<td>1.00</td>
<td>0.03</td>
<td>0.00</td>
</tr>
<tr>
<td>06CV-indus</td>
<td>0.60</td>
<td>1.25</td>
<td>1.60</td>
<td>1.75</td>
<td>0.15</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Later on during the project, and based on results then obtained on experimental material, two compositions were proposed for industrial material. The first one was based on the 0.6%C experimental heats, with modifications to the hardenability and choice of alloying to control grain size.

2.2 WP2: KINETICS OF TRANSFORMATION

Extensive experimental work has been carried out to characterise the transformation kinetics in all alloys designed in the project (Figure 4 et Figure 5). This has lead to two conclusions:

- first, the transformation kinetics can indeed be largely accelerated from the initial requirements of almost one week to slightly over 10 h.
- second, the transformation duration and resulting hardness were shown to depend first on temperature and to a lesser extent (and within the range investigated) on chemical composition.

Figure 4: Time for bainitic transformation and resulting hardness on 1% carbon grades.

Figure 5: Time for bainitic transformation and resulting hardness on 0.6% carbon grades.
2.3 WP3: Development of Heat-Treatments

Dry-bainitising was successfully used to heat-treat a majority of the 0.6% C grades during experiments on laboratory heats. The hardenability of the 1%C grade was shown, during the course of the project, to be considerably less than anticipated from calculations. Thus, and although ALD was later able to adjust the heat-treatment parameters to achieve satisfactory results, a number of heat-treatments on 1%C grades were carried out using salt-baths. Results obtained on actual heat-treated specimens were found to have microstructure and hardness very similar to that measured on dilatometry specimens (WP2).

2.4 WP4: Microstructural Investigations

The aim of the "microstructural investigations" WP was to provide quantitative data such as retained austenite and bainite plate thickness. While initially intended to be carried out only on selected specimens, both retained austenite and bainite lath thickness measurements were eventually carried out on all samples, thus far exceeding the amount of work initially planned.

It was shown that the ratio $V_b/t_b$ (bainite fraction / bainite lath thickness) correlated well with hardness (Figure 6) and yield strength.

![Figure 6: Hardness of different samples vs Vb/tb (bainite fraction / bainite lath thickness).](image)

Retained austenite was surprisingly similar in most of the investigated conditions; with the exception of the high Si grade (1CSi, 2.9% Si), for which the typical austenite content exceeded 30%.

2.5 WP5: Wear Resistance

Wear resistance was evaluated using twin-disc dry rolling-sliding (RS) experiments, and abrasive wear tests. The RS tests demonstrated a far lower specific wear rate for all nanobainitic grades, even when compared to conventional (Si "free") 100Cr6 (Figure 7). This may be related to the higher retained austenite stability, known to be beneficial in contaminated rolling contact fatigue tests [PDa,2000].
Figure 7: Specific wear rate for investigated Nanobain grades and for a number of references provided by LTU. Laboratory heats of Nanobain grades typically exhibited 50% lower wear than conventional material with identical hardness. Industrial heats exhibited even further reduced wear, with, in the case of 1CSi heat-treated at 220°C, almost no detectable wear over the duration of the test.

Abrasive wear performances as evaluated by METSO were also promising with performances on par or above those of the current references for this end-user (Figure 8).

Figure 8: Abrasive wear resistance of Nanobain grades against that of Hardox 500 (0.3C-1.6Mn-1Cr-1.5Ni-0.6Mo, QT).

2.6 WP6: TENSILE AND FATIGUE PROPERTIES

Tensile tests have confirmed that all grades could achieve UTS of 2 GPa and above, with non negligible ductility (Figure 9). A particular surprise however was the discovery of extraordinary ductility in the 1CSi alloy (3 Si wt%), with above 10% uniform elongation for a UTS of 2 GPa. As a measure of comparison, the standard grade 100Cr6 does not exhibit measurable ductility when heat-treated to 2 GPa and above.

Attempts to understand the origin of ductility in most specimens have however been met with only partial success. Indeed, the selected microstructural parameters (retained austenite content, bainite lath thickness), while well correlated with yield strength or hardness, fail to provide any indication of the ductility. This is particularly visible with the results shown in Table 3, where similar parameters lead to
significantly different ductility. Similar results have been obtained for toughness, with a clear difference in results when heat-treating at 220 or 250 °C. This difference was again not seen on microstructural parameters. It is thus likely that retained austenite stability and not quantity is the key to ductility in those microstructures. This is in part the object of further research.

Fatigue properties as measured on laboratory heats were satisfactory on 0.6%C grades with typical fatigue strength at $10^7$ cycles of 0.55-0.6 UTS (after Kt correction) but below expectations on 1%C grades (typically 0.33 UTS after Kt correction). Reasons for this are discussed but not entirely clarified.
2.7 WP7: INDUSTRIAL HEATS AND COMPONENTS

On the basis of experimental results collected on laboratory grades, two industrial heats were manufactured (Table 2). The 06CV industrial material was used to manufacture blades for scrap shear (METSO). Microstructural investigations demonstrated that the alloy design had been successful. Indeed, the component, approximately 80 mm thick, exhibited a homogeneous microstructure throughout after heat-treatment, and its hardness was as expected from initial dilatometry experiments.

After a first trial, performances in the field were reasonably similar to those of the reference material in this context (45NiCrMoV16-6) thus potentially leading to cost savings around 20%.

![Figure 10: Scrap shear and nanobainitic cutting blades after test.](image)

Industrial grades were also tested for fatigue resistance, using both rotation bending tests, notched push-pull specimens and component demonstrators. Once again, the fatigue resistance was satisfactory for the 06CV industrial grades but well below expectations for the 1CSi steel.

2.8 GENERAL CONCLUSIONS

In just over three years, the Nanobain project took the concept of nanostructured bainitic steels from a laboratory experiment to full scale industrial production and testing. A first achievement was to demonstrate that reasonable transformation kinetics could be achieved through tailoring of the alloy hardenability to its application. This was done without the use of expensive alloying elements such as Co, Ni, etc. thus leading to a reasonably inexpensive material. A second achievement was to demonstrate the superior potential of nanostructured carbide-free bainite for abrasive wear applications, and confirm, through industrial testing, that the material was on par with significantly more expensive alloy steels.

Fatigue performance of the 0.6%C material was on par with bainitised 100Cr6 regardless of the manufacturing route. Results achieved on 1CSi were significantly lower and may partly be attributed to poor cleanliness.

Finally, as an unexpected result of tests carried out on high Si material, the highest combination of UTS and elongation was achieved for this type of material.
1. WP1: THEORETICAL DESIGN OF NANOSTRUCTURE BAINITIC STEELS

1.1 OBJECTIVES

This WP had three objectives:
- to propose on the basis of thermodynamics modelling, steel grades that are potentially successful candidates for obtaining the required microstructures
- to improve the fundamental understanding of the reaction kinetics
- to create a tool that will significantly simplify the design of nanostructured bainitic alloys given target mechanical properties and a processing route.

1.2 TASK 1.0 AND 1.1: ALLOY DESIGN

1.2.1 Choice of steel grades

At the project launch meeting, two target components were identified: a high pressure injection component for BOSCH, and a mill lining block for METSO. Both components have significantly different dimensions, the former being typically 100-200 mm long and 20-30 mm in diameter, the latter being 75 mm thick for a length and width of 400x235 mm². Because of its size, METSO’s component is unlikely to be heat-treatable by gas quenching, so that salt-bath quenching will be used instead. On the contrary, BOSCH’s component is amenable to dry bainitising.

As the required mechanical properties differ, and as both components were thought likely to require different hardenabilities, the partners agreed that alloy design would follow the division highlighted in Table 4:

<table>
<thead>
<tr>
<th>Hardenability for Bosch component</th>
<th>Hardenability for Metso’s component</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6% C (target 1600 MPa)</td>
<td>2 heats</td>
</tr>
<tr>
<td>1.0 %C (target 2500 MPa)</td>
<td>2 heats</td>
</tr>
</tbody>
</table>

Table 4: Division of the different heats to be prepared by Ascometal and Sidenor, according to initial planning.

The details of the compositions to be manufactured were the object of WP1.

1.2.2 Estimation of hardenability for METSO's component

It is reminded here that bainitising consists in an austenitisation at a given temperature above AC3 (as would be used for standard quenching), followed by quenching to a temperature above Ms where the component is held for a duration sufficient to achieve the maximum volume fraction of bainite. Thus, a conventional quenching process is not applicable, and salt-bath or interrupted gas-quenching are used instead. In all cases, for the transformation to occur in a controlled manner throughout the component, it is essential that, even at the slowest cooling point of this component, the cooling rate be sufficient to avoid formation of ferrite/pearlite or other transformation products during continuous cooling (upper bainite for example).

Metso’s component being approx. 75 mm thick, it was important to estimate the cooling rate at the centre of the future test piece, to ensure that the hardenability of the designed alloy would be sufficient to avoid formation of ferrite/pearlite. Thus, based on a combination of experimental work and simulation, the cooling rate of this component during salt bath bainitising was estimated to approximately 1.5-2 °C/s. It was thus concluded that the adapted steel grade would be designed not to form ferrite/pearlite for any cooling rate faster than 1.5 °C/s. Details are provided in Appendix A.

1.2.3 Design of adapted steel grades

Based on thermodynamic modelling of the bainite reaction, and with input from the required UTS and hardenability, a total of 9 experimental alloys were proposed (Table 5).
The design was carried out with the following criteria:
- hardenability as per target application requirements
- bainite formation kinetics: as fast as possible, preferably under 10 h
- Ms below 200 °C
- no highly expensive alloying additions such as Co, or detrimental to other properties such as Al (cleanliness)

Details are provided in Appendix B.

1.3 TASK 1.2 AND 1.4: MODEL VALIDATION

1.3.1 Introduction

As detailed later (Appendix E), the kinetics of transformation of all the experimental alloys were determined using dilatometry, together with Ms and Bs for each alloy. The modelling approach is here discussed in view of these results.

1.3.2 Bs and Ms temperatures

The theory underlying the calculations of the maximum temperature at which bainite can form states that bainite nucleation is only possible once the Gibbs energy accompanying the transformation exceeds that required for its nucleation under paraequilibrium conditions (C diffusion), and its diffusionless growth [HKDB, 1981]. The requirement for nucleation has been shown to be well described by a universal nucleation function based on a dislocation mechanism of the kind associated with martensite [HKDB, 1981; HKDB, 2001a; GBO, 1976; CGM, 2004].

Together with the growth condition, the function allows the calculation of the bainite start temperature, Bs, from the knowledge of thermodynamics alone. This theory was used during the alloy design procedure to ensure that transformation temperatures were low or even lower than those for the reference alloys. Further details are provided in Appendix B.

Figure 11 summarises the experimental results, which clearly show that the aim of keeping and even lowering the bainitic transformation range was achieved. The Bs of the new alloys is referred as the maximum tested temperature where bainite was obtained, therefore the exact value of the Bs has not been obtained, but it is not expected to differ much from that reported.
1.3.3 Enhancement of the transformation kinetics

Influence of composition

As mentioned earlier and in Appendix B, the driving force for bainite formation was used as an indicator of kinetics, in agreement with existing theory [HKDB, 2001a]. This has been a success inasmuch as transformation times of 10 h or less have been achieved in many of the investigated conditions, whereas previous reference alloys investigated by CENIM led to far longer transformation durations.

Based on measurement of transformation kinetics, a correlation was sought between overall transformation duration and calculated driving force. As shown in Figure 12, there are significant differences in kinetics at all temperatures investigated. However, although it may be argued that the 0.6%C grades exhibit some degree of correlation between transformation duration and driving force, this is clearly not the case for the higher carbon grades. For the latter, the calculated driving force is nearly identical, although the transformation duration varies by approximately 50%.

There is thus a significant deviation from what the underlying model would have led to expect in terms of differences in transformation kinetics. A number of results obtained by CENIM and co-workers over the course of the project may shed light on the origin of the discrepancies.

Figure 11: Experimental results on the $M_s$ and $B_s$ temperatures for the reference and new alloys. Details for the reference alloys are to be found in Appendix B.

Figure 12: Transformation duration versus driving force at given temperatures for the alloys investigated. Hollow points are for grades based on 0.6%C.
First, analysis of ferrite and austenite content using atom probe tomography (APT) have shown significant changes in the carbon distribution, and in particular a ferrite carbon content above that implicitly expected in the thermodynamics calculation [CGM, 2012a]. Second, the very high dislocation density at the ferrite austenite interface will undoubtedly affect the carbon redistribution, thus again explaining deviation from the underlying assumptions of the kinetics model [FGC, 2011a].

Thus, a thorough revision of the actual understanding of the mechanism of transformation at low temperatures is required before a satisfactory model can be proposed to account for the detailed variations between the grades manufactured in the project.

**Influence of prior austenite grain size (PAGS)**

A second method investigated in the course of the project to accelerate bainite transformation kinetics was to refine PAGS. For this purpose, as detailed in Table 5, two grades were manufactured with additions of small amounts of Nb. This was shown to be effective in reducing PAGS in the 1%C grade (1CNb). This reduction led to a measurable increase in transformation kinetics. It was later realised however, that the manufacturing route for industrial alloys prohibited the use of Nb. Details of the investigation of PAGS and its relationship to bainite transformation kinetics are to be found in Appendix C.

**1.4 TASK 1.3 : PROPOSITION OF COMPOSITIONS FOR INDUSTRIAL MATERIALS**

The aim of task 1.3 was to propose compositions for the manufacture of industrial heats, on the basis of results achieved on laboratory casts.

**1.4.1 06C based grade**

Based on experimental results for the 06CNb grade, in particular toughness and wear resistance results, and after estimation of the required hardenability, the following composition was proposed for the 06C industrial grade (Table 6).

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.65</td>
<td>1.25</td>
<td>1.60</td>
<td>0.02</td>
<td>0.02</td>
<td>1.75</td>
<td>0.15</td>
<td>0.20</td>
<td>0.20</td>
<td>0.03</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Table 6 : Proposed composition for the 06CNb based industrial heat. Nb has actually been replaced by V to avoid formation of primary carbonitrides.

To avoid formation of primary Nb nitrides in large ingot castings, (Appendix D), it was decided not to use niobium in the industrial material.

**1.4.2 1CSi material**

Following the exceptional results achieved on the 1CSi laboratory grade, it was decided to base the 1%C industrial heat on this composition. As results had shown difficulties in heat-treating even small diameters, the Cr content was increased slightly (Table 7).

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.00</td>
<td>0.75</td>
<td>2.50</td>
<td>0.01</td>
<td>0.03</td>
<td>1.00</td>
<td>0.15</td>
<td>0.20</td>
<td>0.003</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Table 7 : Target composition for the 1CSi industrial grade. Cr content was increased compared to the laboratory cast, to achieve slightly higher hardenability.

**1.5 CONCLUSIONS AND ACHIEVEMENTS**

Through the use of theoretical models, WP1 has lead to the design of 9 experimental laboratory grades, with the aim of achieving transformation durations around 10 h, with tensile strength at or above 1600 / 2000 MPa after bainitising at 220 °C or above.

While both objectives were achieved, comparisons between variations in measured transformation kinetics and calculated driving forces did not exhibit a significant correlation. It is thought that the underlying assumptions of the model need revising in the light of recent results.
On the basis of laboratory results, two compositions were proposed for the industrial heats to achieve the objectives of the project.

2. WP2: EXPERIMENTAL ALLOYS AND BAINITE KINETICS

2.1 OBJECTIVES

This WP has two objectives:
- to manufacture appropriate compositions, with the help of the results from task 1.1
- to measure the kinetics of bainite formation in these materials

2.2 TASK 2.1: PRODUCTION OF EXPERIMENTAL MATERIAL

2.2.1 Introduction

Because of the slightly different production methods, laboratory casts manufacture is discussed by partner.

2.2.2 Ascometal

Initial set of laboratory casts

Laboratory steel grades were induction melted under vacuum, then cast into 100x100x200 mm$^3$ rectangular ingots (20 kg). The lower and upper parts of the ingots were then discarded after cutting. The remainder was manually forged into a ~40 mm diameter bar (typical forging temperature 1200-1250 °C). This procedure was applied for all heats except where indicated.

Following technical difficulties which will be detailed below, or because of a need for additional material, Ascometal manufactured a total of 15 heats for the first part of the project. For the sake of clarity, all casts manufactured by Ascometal are presented below (Table 8 and Table 9).

---

<table>
<thead>
<tr>
<th>Ascometal Cast Ref.</th>
<th>Nanobain ref. (target)</th>
<th>C-leco</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
<th>S leco</th>
<th>P</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>1C</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HE33</td>
<td>0.99</td>
<td>1.33</td>
<td>0.69</td>
<td>0.45</td>
<td>0.023</td>
<td>0.001</td>
<td>0.048</td>
<td>0.021</td>
<td>0.10</td>
<td>0.20</td>
<td>0.037</td>
<td>Si below target</td>
<td></td>
</tr>
<tr>
<td>HE36</td>
<td>0.99</td>
<td>1.58</td>
<td>0.76</td>
<td>0.46</td>
<td>0.022</td>
<td>0.001</td>
<td>0.047</td>
<td>0.021</td>
<td>0.10</td>
<td>0.20</td>
<td>0.047</td>
<td></td>
<td></td>
</tr>
<tr>
<td>IE29</td>
<td>1.08</td>
<td>1.57</td>
<td>0.77</td>
<td>0.45</td>
<td>0.020</td>
<td>0.001</td>
<td>0.036</td>
<td>0.021</td>
<td>0.10</td>
<td>0.20</td>
<td>0.043</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>1CNb</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HE34</td>
<td>1.00</td>
<td>1.29</td>
<td>0.71</td>
<td>0.45</td>
<td>0.023</td>
<td>0.019</td>
<td>0.047</td>
<td>0.020</td>
<td>0.10</td>
<td>0.20</td>
<td>0.043</td>
<td>Si below target</td>
<td></td>
</tr>
<tr>
<td>HE37</td>
<td>1.02</td>
<td>1.53</td>
<td>0.76</td>
<td>0.46</td>
<td>0.022</td>
<td>0.018</td>
<td>0.049</td>
<td>0.022</td>
<td>0.10</td>
<td>0.20</td>
<td>0.051</td>
<td></td>
<td></td>
</tr>
<tr>
<td>IE27</td>
<td>1.05</td>
<td>1.62</td>
<td>0.75</td>
<td>0.51</td>
<td>0.029</td>
<td>0.022</td>
<td>0.049</td>
<td>0.020</td>
<td>0.10</td>
<td>0.20</td>
<td>0.024</td>
<td></td>
<td></td>
</tr>
<tr>
<td>-</td>
<td>1CMo</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HE35</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>0.100</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 8: Chemical composition of 1% C laboratory heats manufactured by Ascometal, in wt%. Italic font indicates LECO analysis results. All other results obtained by OES.

As shown in Table 8, both initial laboratory heats with 1% C had to be produced twice as Si yield was not known for these compositions. The existing data led to an overestimation of the expected final Si content and therefore an actual content below target.

The chemical compositions for all 06Cx grades manufactured by Ascometal are shown in Table 9. Most of the 06Cx grades had to be manufactured more than once for a variety of reasons: in the first series of laboratory heats, Mo content was considered too low for the target in the HE39 cast. HE38 and IE01 were considered acceptable and used for CCT/TTT determination.
Table 9: Chemical composition of 06Cx laboratory heats manufactured by Ascometal, in wt%. Results obtained by OES except for C, S by LECO.

However, further investigations of the as-forged microstructure in HE38 and IE01 revealed a large number of microcracks due to the high hardenability of the 06Cx materials. All 06Cx grades manufactured were found to be air-hardenable for a diameter of 40 mm, with microstructures exhibiting about 5% pearlite/95% martensite in the 06CCr grade and fully martensitic in the 06CMo grade. Because of the presence of microcracking, heats HE38 and IE01 were only used for dilatometric measurements, for which internal soundness is not critical. However, two additional heats had to be manufactured for production of mechanical testing specimens. Changes in the base material used for laboratory casts led to three casts being discarded before yield could be properly estimated. The resulting heats, IE17 and IE15 were forged as 40 mm diameter bars, but were immediately placed into a furnace (650 °C) after forging and allowed to cool slowly in this furnace.

Additional laboratory heats

At a later stage of the project, three additional laboratory casts were manufactured. The first two with a composition identical to 06C and 08C to provide additional material for fatigue testing. The third one to validate the intended industrial heat composition in terms of hardenability and hot ductility. Details are provided in Table 10.

Table 10: Chemical compositions of three additional heats manufactured by Ascometal during the second part of the project.

2.2.3 Sidenor

Initial set of laboratory casts

Sidenor manufactured four casts into ingots of about 35 kg. Ingots were forged into bars of diameter 50 mm. The composition of the laboratory heats are shown in Table 11.
<table>
<thead>
<tr>
<th>Sidenor Cast ref.</th>
<th>Nanobain ref. (target)</th>
<th>C-leco</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
<th>S leco</th>
<th>P</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>1CSi</td>
<td>1,00</td>
<td>2,50</td>
<td>0,75</td>
<td>0,50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>711</td>
<td></td>
<td>0,98</td>
<td>2,90</td>
<td>0,77</td>
<td>0,45</td>
<td>0,004</td>
<td>-</td>
<td>0,014</td>
<td>0,016</td>
<td>0,16</td>
<td>0,21</td>
<td>0,040</td>
<td>two heats</td>
</tr>
<tr>
<td>08C</td>
<td></td>
<td>0,80</td>
<td>1,50</td>
<td>0,75</td>
<td>0,50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>703</td>
<td></td>
<td>0,90</td>
<td>1,65</td>
<td>0,79</td>
<td>0,48</td>
<td>0,004</td>
<td>0,003</td>
<td>0,016</td>
<td>0,015</td>
<td>0,17</td>
<td>0,20</td>
<td>0,020</td>
<td></td>
</tr>
<tr>
<td>06C</td>
<td></td>
<td>0,60</td>
<td>1,50</td>
<td>1,25</td>
<td>1,50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>706</td>
<td></td>
<td>0,68</td>
<td>1,60</td>
<td>1,25</td>
<td>1,50</td>
<td>0,003</td>
<td>0,003</td>
<td>0,016</td>
<td>0,012</td>
<td>0,16</td>
<td>0,20</td>
<td>0,012</td>
<td></td>
</tr>
<tr>
<td>06CNb</td>
<td></td>
<td>0,60</td>
<td>1,50</td>
<td>1,25</td>
<td>1,50</td>
<td>0,030</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>707</td>
<td></td>
<td>0,64</td>
<td>1,60</td>
<td>1,27</td>
<td>1,50</td>
<td>0,003</td>
<td>0,035</td>
<td>0,016</td>
<td>0,013</td>
<td>0,16</td>
<td>0,20</td>
<td>0,012</td>
<td></td>
</tr>
</tbody>
</table>

Table 11: Chemical composition of laboratory heats manufactured by Sidenor, in wt%. Results obtained by OES except for C, S by LECO.

The 1CSi heat had to be cast again due to problems during hot forging. In the second attempt, the forging temperature for that particular grade was kept below 1025 °C in order to avoid burning. A piece of as-forged 1CSi steel was sent to CENIM for CCT/TTT characterisation. All forged bars were soft cooled to avoid stress cracks and then annealed at 700 °C for 2 hours to make specimen machining easier.

Additional heat
In order to provide sufficient material for fatigue testing, an additional laboratory heat of 1CSi was manufactured during the second part of the project. However, as this exhibited numerous problems (cracking, graphite formation) due to the excess Si, it was decided to carry out the fatigue tests on the equivalent industrial material. The laboratory grade was thus only used for evaluation of the hot ductility.

<table>
<thead>
<tr>
<th>Sidenor Cast ref.</th>
<th>Nanobain ref. (target)</th>
<th>C-leco</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
<th>S leco</th>
<th>P</th>
<th>Ni</th>
<th>Cu</th>
<th>Al</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>-</td>
<td>1CSi</td>
<td>1,00</td>
<td>2,50</td>
<td>0,75</td>
<td>0,50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>724</td>
<td></td>
<td>0,98</td>
<td>3,27</td>
<td>0,77</td>
<td>0,47</td>
<td>0,009</td>
<td>-</td>
<td>0,016</td>
<td>0,018</td>
<td>0,15</td>
<td>0,20</td>
<td>0,039</td>
<td></td>
</tr>
</tbody>
</table>

Table 12: Chemical composition (wt%) of the additional 1CSi grade manufactured in the laboratory by Sidenor. Results obtained by OES except for C, S by LECO.

2.2.4 Conclusions
Numerous difficulties were met, first with the Si yield leading to poorly control Si content, second with the cracking occurring during natural cooling of the small diameter bars used in laboratory experiments, and finally with unexpected difficulties such as formation of graphite. A total of more than 20 laboratory casts were manufactured during the project.

2.3 TASK 2.2: KINETICS OF LOW-TEMPERATURE BAINITE

2.3.1 Introduction
The purpose of Task 2.3 was to investigate the kinetics of low temperature bainite formation in all manufactured grades, together with the hardness of the resulting product. It is reminded here that bainitising heat-treatments consist in austenitising at temperatures above Ac3 (or Acm for hypereutectoid steels) followed by "rapid" cooling to the required transformation temperature T_B (Figure 13). How "rapid" the cooling needs to be depends on the hardenability of the steel grade, since the only requirement is to avoid transformation during cooling.
The first points worthy of investigation are thus:
- Ac1, Ac3/Acm to determine suitable austenitising conditions
- Ms to ensure that bainitising can be carried out above this temperature
- The critical cooling rate below which transformation occurs during continuous cooling, for which continuous-cooling-temperature (CCT) diagrams are required

These points were identified by dilatometry (further details can be found in Appendix E).

In a second step, the duration of bainite transformation was determined for all alloys and for a range of transformation temperatures. This was also carried out using dilatometry. Further details are provided in Appendix E.

### 2.3.2 Characteristic transformation temperatures

The characteristic transformation temperatures and critical cooling rate are shown in Table 13.

<table>
<thead>
<tr>
<th></th>
<th>AC1 / °C</th>
<th>AC3 / °C</th>
<th>Ms / °C</th>
<th>Critical CR / °C/s</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>742</td>
<td>880</td>
<td>130</td>
<td>20</td>
</tr>
<tr>
<td>1CMo</td>
<td>742</td>
<td>880</td>
<td>130</td>
<td>15</td>
</tr>
<tr>
<td>1CNb</td>
<td>741</td>
<td>881</td>
<td>123</td>
<td>20</td>
</tr>
<tr>
<td>1CSI</td>
<td>769</td>
<td>893</td>
<td>165</td>
<td>20</td>
</tr>
<tr>
<td>06C</td>
<td>770</td>
<td>838</td>
<td>217</td>
<td>5</td>
</tr>
<tr>
<td>06CMo</td>
<td>795</td>
<td>853</td>
<td>220</td>
<td>3</td>
</tr>
<tr>
<td>06CNb</td>
<td>766</td>
<td>840</td>
<td>207</td>
<td>5</td>
</tr>
<tr>
<td>06CCr</td>
<td>808</td>
<td>872</td>
<td>240</td>
<td>5</td>
</tr>
<tr>
<td>08C</td>
<td>758</td>
<td>808</td>
<td>165</td>
<td>13</td>
</tr>
</tbody>
</table>

Table 13: Characteristic transformation temperatures for all laboratory alloys investigated. Ac3 must be understood as Acm for 1C grades.

From the measurements of Ac3, it was decided that a temperature of 900 °C would be sufficient for the austenitisation of all 06C grades. A temperature of 950 °C was used for all other grades.

The martensite start temperatures (Ms) was typically well below 200 °C for the 1C grades thus allowing bainitising at very low temperatures. In 06C grades however, bainitising was limited to temperatures above 220 °C or even 240 °C in one case.
Finally, the critical cooling rate for 1C grades was well below that expected from calculation (typically 20 °C/s against an estimated 0.35 °C/s by calculation). This was nevertheless deemed sufficient for Bosch's application and manufacturing route. The critical cooling rate for 06C grades was also slightly below that required for Metso's component (see WP1), with typically 3-5 °C/s against a requirement of 1.5 °C/s. This prompted significant adjustments when manufacturing the corresponding industrial heats. The supporting CCT diagrams are shown in Appendix E.

2.3.3 Kinetics of transformation and resulting hardness

2.3.3.1 1C grades

Transformation durations at the different temperatures investigated are shown in Figure 14. The results for the 08C grade, for which hardenability is similar to that of the 1C, 1CMo, 1CNb and 1CSi grades, are also shown for comparison. All hardness measures were carried out with a 30 kg load unless otherwise indicated.

![Figure 14](image1)

**Figure 14**: Time to fully transform the different 1Cx grades as a function of temperatures. Times at 200 (and to a smaller extent at 220 °C) are often estimated from partially transformed specimens, as the duration of the experiment becomes prohibitive.

![Figure 15](image2)

**Figure 15**: Hardness as a function of transformation temperature for the different 1Cx grades. Values below 240 °C must be interpreted with care.

As can be seen in Figure 15, the hardness obtained on the 1Cx is virtually identical for all four grades from 220 to 350 °C. At 200 °C, dispersion in results are mainly a result of varying degrees of transformation since the time allowed in the dilatometer was seldom sufficient to reach the end of the
The 08C grade appears particularly interesting as it offers the same hardness as the 1C grades at temperatures below 240 °C and a higher hardness above that temperature. It is worth noting that, at 220 °C, the 08C grade also transforms significantly faster than the 1Cx grades (Figure 14).

### 2.3.3.2 06C grades

![Figure 16: Time to fully transform the different 06Cx grades as a function of temperatures.](image)

Bainite reaction durations in 06Cx and 08C grades are shown in Figure 16. For 06CCr and 06CMo, the Ms temperature of 220-240 °C prevented the use of transformation temperatures lower than 260 °C.

![Figure 17: Hardness as a function of transformation temperature for the different 06Cx grades.](image)

As shown in Figure 17, the hardness as a function of transformation temperature follows a trend similar to that observed on 1Cx grades above and at 260 °C, but is typically 50HV lower below and at 240 °C.

### 2.4 CONCLUSIONS AND ACHIEVEMENTS

The characteristic transformation temperatures and hardenability were investigated for all alloys and led to the definition of suitable conditions for heat-treating of specimens.
The kinetics of bainite formation in all experimental alloys were measured and shown to be primarily dependent on temperature rather than alloy composition, as discussed in WP1.

Although some delays were experienced during this work package, no major deviation occurred in content or schedule.

3. **WP3: DEVELOPMENT OF HEAT-TREATMENTS**

3.1 **OBJECTIVES**

The objective of this work package was to develop and carry out all the required heat-treatments using a newly developed, more environmental-friendly bainitising process.

3.2 **TASK 3.1 AND 3.2 : HEAT-TREATMENTS, VALIDATION**

3.2.1 **Introduction**

Details of the specimens and heat-treatment to be carried out were established following work carried out in WP2 (Table 14).

<table>
<thead>
<tr>
<th>Grade</th>
<th>Samples</th>
<th>Temperature / °C</th>
<th>Critical cooling rate / °C.s⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>06C</td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>250</td>
<td>5</td>
</tr>
<tr>
<td>06Cr</td>
<td>TENS, MET</td>
<td>250</td>
<td>5</td>
</tr>
<tr>
<td>06CMo</td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>250</td>
<td>3</td>
</tr>
<tr>
<td>06CNb</td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>220</td>
<td>5</td>
</tr>
<tr>
<td>08C</td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>220</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>250</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>270</td>
<td>13</td>
</tr>
<tr>
<td>1C</td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>250</td>
<td>20</td>
</tr>
<tr>
<td>1CMo</td>
<td>TENS, MET</td>
<td>200</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>TENS, MET</td>
<td>220</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>TENS, MET</td>
<td>250</td>
<td>20</td>
</tr>
<tr>
<td>1Nb</td>
<td>TENS, MET, FAT</td>
<td>220</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>TENS, MET</td>
<td>240</td>
<td>15</td>
</tr>
<tr>
<td>1Si</td>
<td>TENS, MET</td>
<td>220</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>TENS, KCV, RS, SCF, HPA, MET</td>
<td>250</td>
<td>20</td>
</tr>
</tbody>
</table>

Table 14: Materials and heat-treatment schedules for all mechanical and metallography specimens.

The following references are used to identify the different kinds of mechanical testing specimens (further details in WP4-6):

- TENS: tensile specimen
- KCV: V-notch Charpy impact specimen
- HPA: high-pressure abrasion testing specimen
- SCF: standing contact fatigue specimen
- RS: rolling-sliding wear specimen
- MET: metallography specimen

With the exception of MET specimens (which are simply coupons), all samples were rough machined before heat-treatment, work being distributed according to the proposal.

According to results obtained from WP2, the conditions for heat-treatment were defined as per Table 15. The table shows “connected” values with “/” e.g. a transformation temperature of 200 °C corresponds with a transformation time of 40 h, etc. Table 15 also shows some of the parameters used at the plant at ALD. The austenizing time was used for technical reasons in order to provide a complete heating of load derived by experience. The same applied for the quench time. Comprehensive experience showed that a quench time of 300 s is sufficient for cooling down the load to the targeted transformation temperature.
### Table 15: Heat-treatment parameters for dry-bainitising.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Austenizing temperature / °C</th>
<th>Austenizing time / min</th>
<th>Quench time / s</th>
<th>Transformation temperature / °C</th>
<th>Transformation time / h</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>950</td>
<td>65</td>
<td>300</td>
<td>250</td>
<td>12</td>
</tr>
<tr>
<td>1CMo</td>
<td>950</td>
<td>65</td>
<td>300</td>
<td>200 / 220 / 250</td>
<td>40 / 20 / 12</td>
</tr>
<tr>
<td>1CNb</td>
<td>950</td>
<td>65</td>
<td>300</td>
<td>220 / 240</td>
<td>20 / 15</td>
</tr>
<tr>
<td>1CSi</td>
<td>950</td>
<td>65</td>
<td>300</td>
<td>220 / 250</td>
<td>20 / 12</td>
</tr>
<tr>
<td>08C</td>
<td>890</td>
<td>65</td>
<td>300</td>
<td>220 / 250 / 270</td>
<td>15 / 12 / 7</td>
</tr>
<tr>
<td>06C</td>
<td>890</td>
<td>65</td>
<td>300</td>
<td>250</td>
<td>12</td>
</tr>
<tr>
<td>06CMo</td>
<td>890</td>
<td>65</td>
<td>300</td>
<td>250</td>
<td>12</td>
</tr>
<tr>
<td>06Cr</td>
<td>890</td>
<td>65</td>
<td>300</td>
<td>250</td>
<td>12</td>
</tr>
<tr>
<td>06CNb</td>
<td>890</td>
<td>65</td>
<td>300</td>
<td>220 / 250</td>
<td>22 / 12</td>
</tr>
</tbody>
</table>

#### 3.2.2 Trials

In order to achieve the necessary cooling time a gas speed enhancing device was used inside the quench cell. Thus the gas speed was multiplied. Corresponding with that device, the load size itself was limited to approx. 390x170mm initially. The quench chamber was equipped with various thermocouples for controlling the gas temperature on different positions. These sensors’ data were all logged for later analysis together with the data of the quench pressure. By analyzing these collected data it was possible to check if any process deviation took place.

![Heat-treatment plant](image)

**Figure 18**: Heat-treatment plant.

In preparation of the Nanobain-trials, extensive research was conducted on assuring the right cooling rate. Therefore several bolts with varying diameters (10 to 50 mm) made of 100Cr6 where heated up, austenitized according to the parameters above and quenched. While quenching, the load was monitored by a thermographic camera providing the temperature on the surface of the bolt as raw data and additionally as an optical video. With this procedure it was possible to first ensure that no part of the load was dropping below Ms-temperature and second to retrieve data on the cooling rate. As most of the samples of the Nanobain-project did have similar diameters and weights compared to the bolts, a reliable comparison of the cooling rate was possible.
Prior to the heat treatment all samples in one load were weighted, measured and photographed. Whenever feasible, samples were collected in one load for heat-treatment. Figure 20 shows examples of typical heat-treatment loads. MET-samples were always standing so as to expose the outer surface to the gas flow (diameter along the vertical direction), all other were in flat position and softly fixed by small wire. For some grades, the amount of samples per trial made it necessary to split up the samples in several batches.

The quench pressure for all loads was 4.9 bar, which was the maximum pressure within the safety margins of the plant taking into consideration the gas expansion while quenching.

The heating and austenizing phase of the trials went without any problems. In all cases the gas inlet speed was less than 4 sec (to reach the final pressure). Also most of the quenches were performed without any problems. However there was one case, where the targeted final quench pressure was not reached.

First results of the 1CNb-220C-samples showed, that the cooling rate for this steel grade was not reached (see below). Therefore the gas speed enhancing device inside the quench chamber was changed end of October '09 thus doubling the gas speed.

### 3.2.3 Results

After the first heat-treatment trials, it was noted that the cooling rate of the 1C-grades was not sufficient for avoiding pearlite formation. This was especially true for the MET-samples which were the most “compact” and heavy ones. On the 1CNb-220C-TENS samples however a microstructure was identified which corresponds to a cooling rate of around 15 °C/s.
It was therefore decided to change to the smaller load size of 180x70 mm (see Figure 20; right) with doubled gas speed. Further it was decided to heat treat all critical samples in salt bath at ASCOMETAL. The main reasons were to avoid loss of samples and time within the project schedule. Critical samples were all samples with a critical cooling rate around 15-20 °C/s (ie, 1C* and 08C grades) and with following examinations on specific properties which were important for the decision on the industrial heat (WP 7). The heat treatment parameters of ASCOMETAL were as per Table 16:

<table>
<thead>
<tr>
<th>Grade</th>
<th>Austenizing temperature / °C</th>
<th>Austenizing time / min</th>
<th>Transformation temperature / °C</th>
<th>Transformation time / h</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>950</td>
<td>60</td>
<td>250</td>
<td>16</td>
</tr>
<tr>
<td>1CMo</td>
<td>950</td>
<td>60</td>
<td>200/220/250</td>
<td>64 / 22 / 16</td>
</tr>
<tr>
<td>1CNb</td>
<td>950</td>
<td>60</td>
<td>220/240</td>
<td>22 / 16</td>
</tr>
<tr>
<td>1CSI</td>
<td>950</td>
<td>60</td>
<td>220/250</td>
<td>22 / 16</td>
</tr>
<tr>
<td>08C</td>
<td>890</td>
<td>60</td>
<td>220/250/270</td>
<td>22 / 16 / 7</td>
</tr>
</tbody>
</table>

Table 16 : Heat treatment parameters used for salt bath bainitising.

In comparison to the parameters of the dry bainitising process, slightly longer transformation times were applied. Reasons for this were mainly uncertainties about the end of the transformation and the identified transformation kinetics (see WP 1 and 4). The extension to 64 hours (instead of 40) was due to technical reasons and not heat treatment related.

3.2.4 Validation

During the ongoing heat treatment, several samples of different grades were examined. With the additional care taken to ensure sufficient cooling rate either by adapting gas flow or by use of salt-bath quenching, it was possible to avoid pearlite formation in all cases. In additional, the Ms-temperature was never reached.

The following hardness were measured after heat-treatment (Table 17):

<table>
<thead>
<tr>
<th>Grade</th>
<th>200 °C</th>
<th>220 °C</th>
<th>240 °C</th>
<th>250 °C</th>
<th>270 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>751 HV</td>
<td>707 HV</td>
<td></td>
<td>652 HV</td>
<td></td>
</tr>
<tr>
<td>1CMo</td>
<td>714 HV</td>
<td>691 HV</td>
<td></td>
<td>659 HV</td>
<td></td>
</tr>
<tr>
<td>1CNb</td>
<td>650 HV</td>
<td>625 HV</td>
<td></td>
<td>625 HV</td>
<td></td>
</tr>
<tr>
<td>08C</td>
<td>710 HV</td>
<td></td>
<td></td>
<td>659 HV</td>
<td>615 HV</td>
</tr>
<tr>
<td>0.6CMo</td>
<td></td>
<td></td>
<td></td>
<td>602 HV</td>
<td></td>
</tr>
<tr>
<td>0.6CCr</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>589 HV</td>
</tr>
</tbody>
</table>

Table 17 : Hardness of the salt bath bainitized samples (30kg load).

The examinations of the samples treated differently revealed however similar properties which are corresponding to the predictions of the CCT-diagrams of WP2. Judging from the hardness results of the grades with lower carbon content it can be concluded that the dry bainitising process was successful.

However for the grades with higher carbon content, it was difficult using the dry bainitising process to achieve the critical cooling rate and thus the corresponding hardness values. This is due to technical limitations – mainly the quench pressure – and not to the process itself. It has to be pointed out, that the MET-samples are the most massive samples, whereas the TENS or FAT-samples are much smaller in weight and diameter. A hint to that direction was one hardness measurement of 720 HV (30kg load) on a 1CMo-220-TENS-sample. This corresponds to a cooling rate of 15 to 20°C/s and demonstrates the principal functionality of the dry bainitising process. It remains to be investigated if other (smaller) samples with high carbon grades show the same hardness and microstructural properties.
3.3 CONCLUSIONS AND ACHIEVEMENTS

The following conclusions can be presented on the work carried out in WP 3:

- Dry bainitising of 1Cx grades: the hardenability of 1Cx grades is such that pearlite was formed on the largest samples (MET) during the first series of dry bainitising. As a precautionary measure, all 1Cx samples were therefore heat-treated in salt-bath at ASCOMETAL. Nevertheless, hardenability of 1Cx grades appeared sufficient for small diameter samples (impact and tensile specimens) with the initial setup. After modification to increase gas flow, even larger samples could be successfully heat-treated.

- In the first part of the project, heat-treatments were carried out successfully and lead to hardness values similar to those expected, as will be further detailed in WP4.

- During the second part of the project, the dry bainitising furnace prototype at ALD ceased functioning and was decommissioned. Therefore, all additional tests (tensile and fatigue tests carried out on industrial heats, a number of fatigue tests on laboratory casts and bainitising of Bosch's component demonstrators) were carried out using salt-bath at Ascometal or Bosch.

4. WP4: MICROSTRUCTURAL INVESTIGATIONS

4.1 OBJECTIVES

This WP had two objectives:
- to quantify the relative amounts of the different microstructures present in the heat-treated samples obtained in WP2;
- to investigate additional aspects of the microstructure (e.g. the bainite lath thickness and morphology) in order to provide a basis for understanding the mechanical properties.

4.2 TASK 4.1 AND 4.2: GENERAL AND FINE MICROSTRUCTURAL CHARACTERISATION

4.2.1 Introduction

It is reminded here that WP4 was concerned with metallographic characterisation of specimens issued from laboratory casts. Additional characterisations were carried out on specimens issued from industrial heats, these are discussed in WP7.

4.2.2 Experimental procedures

Qualitative metallography

All metallography was carried out using standard specimen preparation methods. Samples were etched in 2 or 4% Nital prior to examination in light optical microscopy (LOM) or scanning electron microscopy (SEM).

X-ray analysis of retained austenite

Determination of retained austenite content using X-ray analysis was carried out at CENIM, ASCOMETAL or LTU. Specimens were electro-polished to avoid alteration of the retained austenite content through surface deformation, as may occur during conventional grinding and polishing processes. Details of the estimation method varied from one laboratory to another, and a round robin was therefore carried out at the onset of the project on two specimens with microstructures similar to those examined in the course of the project. The results showed discrepancies below 1.5%, which was lower than the uncertainty associated with possible texturing effects.

Measurements of bainite lath thickness

Bainite lath thickness was estimated on SEM micrographs after a light Nital etch. A typical micrograph is shown in Figure 21.
Measurements were corrected by $2/\pi$ to account for the effect of section angle compared to lath orientation. Measurements were carried out until the cumulated average was found to reach a stable value. This typically required more than 100 measurements (Figure 22).

4.3 RESULTS

4.3.1 Specimens investigated

All specimens detailed in Table 14 were investigated for general microstructure, bainite lath thickness and retained austenite content. In addition, retained austenite content was measured on some of the dilatometry specimens from WP2.

4.3.2 General observations

All specimens investigated and used in further experiments exhibited mixtures of extremely fine carbide free bainite and retained austenite (see for example Figure 21). Some specimens contained small fractions of pearlite, which was attributed to insufficient cooling rate between austenitising and bainitising. These specimens were not used and heat-treatment was repeated as required to achieve the expected microstructure. The presence of martensite was never observed, as expected from the low Ms temperature of the bulk material, rapidly depressed below room temperature as the transformation progresses.

4.3.3 Retained austenite on dilatometry specimens

A number of dilatometry specimens were investigated for retained austenite content. Results are detailed in Table 18.
<table>
<thead>
<tr>
<th></th>
<th>Transformation temperature / °C</th>
<th>Heat-treatment duration / h</th>
<th>Estimated completion time / h</th>
<th>Measure residual austenite / %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>240</td>
<td>10</td>
<td>11</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>260</td>
<td>7</td>
<td>6.5</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>5</td>
<td>4</td>
<td>27</td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>3</td>
<td>3</td>
<td>53</td>
</tr>
<tr>
<td>1CMo</td>
<td>220</td>
<td>15</td>
<td>18</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td>240</td>
<td>10</td>
<td>10.5</td>
<td>13</td>
</tr>
<tr>
<td></td>
<td>260</td>
<td>7</td>
<td>6</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>5</td>
<td>4.5</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>3</td>
<td>3</td>
<td>49</td>
</tr>
<tr>
<td>1CNb</td>
<td>240</td>
<td>10</td>
<td>10.5</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>260</td>
<td>10</td>
<td>5.5</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>5</td>
<td>3.8</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>350</td>
<td>3</td>
<td>2</td>
<td>49</td>
</tr>
</tbody>
</table>

Table 18: Retained austenite as estimated by X-ray diffraction using dilatometry specimens.

As shown in Figure 23, an unexpected tendency for increase in retained austenite content at the lowest transformation temperature is identified, which is contrary to what would be expected from the theory of bainite transformation [HKDB, 2001a].

![Figure 23](image)

**Figure 23**: Evolution of retained austenite content as a function of bainitising temperature for some 1%C grades, as estimated on dilatometry specimens.

### 4.3.4 Microstructural parameters on heat-treated specimens

The results of the investigation are summarized in Table 19. In a number of cases, investigations were carried out not only on the metallography specimens, but also on the tensile, toughness or RCF specimens, particularly when there was a doubt as to the success of the heat-treatment. Only those specimens whose microstructure had been confirmed to be as expected (bainite + retained austenite) were used for further investigation.
In general, the hardness achieved after salt-bath heat-treatments of mechanical testing specimens was in excellent agreement with that measured on dilatometry samples (WP2). Exceptions concerned those dilatometry cycles where the duration had been insufficient to ensure ending of the bainitic reaction.

As shown in Table 19, the bainite lath thickness exhibited surprisingly little variation with heat-treatment temperature, and appeared to be more strongly correlated to carbon content.

Regarding retained austenite content, comparison between results achieved on specimens of 1CMo heat-treated at 200, 220 and 250 °C do not confirm the 'abnormal' trend identified on dilatometry specimens. This trend however is still present for 1CNb and some of the 06Cx grades. While this may be due to an extremely sluggish transformation rate at low temperature (and therefore insufficient time at transformation temperature), it is also likely that this is simply a consequence of the natural dispersion of the measurement.

In contrast, the 1CSi grades exhibited significantly higher retained austenite content at all temperatures. As will be discussed later, the current knowledge regarding the action of Si in the context of bainite formation does not allow explanation of this result.

### 4.4 CONCLUSIONS AND ACHIEVEMENTS

A full characterisation of all mechanical testing specimens was carried out in WP4. Retained austenite content together with bainite lath thickness were estimated for over 30 specimens (Table 18 only highlighting the results for one condition and successful heat-treatments). Two noticeable results can be highlighted:

- the bainite lath thickness was found to vary relatively little with temperature for a given grade, and for the range of temperatures investigated; on the contrary, this parameter appeared to be related to carbon content, the 0.6% C grades systematically exhibiting coarser bainite laths.
- similarly, variations in retained austenite content for a given grade were found to barely if at all exceed the measurement uncertainty, within the range of temperatures investigated (this is not the case for dilatometry specimens for which temperatures outside of the range for nanostructures bainitic grades were investigated). The retained austenite content of the high Si grade (1CSi) was significantly higher than that of any other alloy in the present study.
5. WP5: WEAR RESISTANCE OF NANOBAINITIC STEELS

5.1 OBJECTIVES AND INTRODUCTION

This work package had two objectives:
- to establish the wear properties of the various alloys
- with WP4, to establish a correlation between microstructural features and wear resistance

The investigation of wear resistance of nanobainitic steels has been carried out using rolling-sliding wear, high stress abrasion wear and Charpy-V impact toughness tests. These methods were selected because the rolling-sliding wear test can simulate the rolling contacts in many different applications, and because the combination of high abrasive wear resistance and impact toughness is a key factor in many industrial applications of METSO.

The nanobainitic steel grades have been compared with different traditional steels. Reference materials used in the rolling-sliding wear tests were 100Cr6, pearlitic CrMo8C6, bainitic CrMo8C5 and traditional spring steels austempered at different temperatures (see Table 20). Reference steels used in the high stress abrasion wear and Charpy-V toughness tests, made by Metso, were the hot-rolled steel Hardox 500, Metso’s tool steel grade LBG2, bainitic CrMo8C5 and pearlitic CrMo8C6.

5.2 TASK 5.1, 5.4 AND 5.5: ROLLING-SLIDING TESTS, INFLUENCE OF MICROSTRUCTURE

5.2.1 Rolling-sliding test method and materials

A detailed description of the test method and conditions is provided in Appendix F. Tests were carried out in all conditions detailed in Table 14 (for which RS specimens were present). Three additional conditions were obtained on conventional (Si "free") 100Cr6 steel (AISI 52100), which are worth detailing.

The first condition, referred to as 100Cr6-std corresponds to the material as used by BOSCH for industrial production. This is austenitised above Ac1 but below Acm. Because 100Cr6 is spheroidised for soft machining operations, the microstructure at the end of austenitising consists in austenite and relatively coarse globular carbides. During bainitising, precipitation of fine carbides is also possible due to the absence of Si. There are thus two microstructural differences when compared to currently investigated alloys (presence of globular carbides, absence of significant retained austenite content since precipitation is possible during the bainite transformation). To generate microstructural conditions closer to those investigated, the 100Cr6 steel was also heat-treated with an austenitising temperature of 950 °C. This temperature ensures that no significant amount of globular carbides is left at the end of the austenitisation (as is the case for Nanobain alloys). Finally, to obtain two levels of hardness in 100Cr6 austenitised at 950 °C, this grade was bainitised at 250 and at 300 °C. Hardness values of approximately 710 and 600 HV respectively were thus achieved. These heat-treatments are summarised in Table 21.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Microstructure</th>
<th>Elements / wt%</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>40CrMo8</td>
<td>Bainite</td>
<td>C 0.09, Si 0.5, Mn 0.9, Cr 2.02, Ni 0.43</td>
<td>Also denoted CrMo8C5</td>
</tr>
<tr>
<td>60CrSiMo9</td>
<td>Pearlite</td>
<td>C 0.61, Si 0.93, Mn 0.62, Cr 2.19, Ni 0.45</td>
<td>Also denoted CrMo8C6</td>
</tr>
<tr>
<td>60SiCr7</td>
<td>Varied</td>
<td>C 0.61, Si 1.72, Mn 0.75, Cr 0.35, Ni 0.12, Mo 0.04</td>
<td>Standard spring steel</td>
</tr>
<tr>
<td>100Cr6</td>
<td>Varied</td>
<td>C 1.0, Si 0.25, Mn 0.75, Cr 1.05, Ni 0.2</td>
<td>Standard bearing steel</td>
</tr>
<tr>
<td>42CrMo4</td>
<td>Varied</td>
<td>C 0.42, Si 0.25, Mn 0.75, Cr 1.05, Ni 0.2</td>
<td>Standard forging steel</td>
</tr>
</tbody>
</table>

Table 20: Composition of additional LTU references.

A number of additional references investigated in previous LTU work were used for comparison purposes. The composition of the different grades is detailed in Table 20 and the conditions in which they were tested are summarised in Table 21. Bainitising the spring steel 60SiCr7 will also lead to a carbide-free microstructure of bainite and retained austenite. However, the alloy content being lower than that of the
0.6 \%C investigated in the present study, the Ms temperature is significantly above 250 °C, and the result of bainitising at this temperature is thus a mixture of tempered martensite and bainite.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Reference</th>
<th>Heat-treatment</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>40CrSi8</td>
<td>40CrSi8</td>
<td>undisclosed, hardness about 400 HV</td>
<td><em>&quot;Conventional&quot;</em> bainite</td>
</tr>
<tr>
<td>60CrSi9</td>
<td>60CrSi9</td>
<td>undisclosed, hardness about 300 HV</td>
<td>Pearlite</td>
</tr>
<tr>
<td>60SiCr7</td>
<td>60SiCr7-250</td>
<td>Austenitising 860°C, bainitising at 250 °C</td>
<td>Carbide free bainite + retained austenite + tempered martensite</td>
</tr>
<tr>
<td></td>
<td>60SiCr7-300</td>
<td>Austenitising 860°C, bainitising at 300 °C</td>
<td>Carbide free bainite + retained austenite</td>
</tr>
<tr>
<td></td>
<td>60SiCr7-350</td>
<td>Austenitising 860°C, bainitising at 350 °C</td>
<td>Carbide free bainite + retained austenite</td>
</tr>
<tr>
<td></td>
<td>60SiCr7-N</td>
<td>Austenitising 860 °C, natural cooling (normalising)</td>
<td>Pearlite</td>
</tr>
<tr>
<td>100Cr6</td>
<td>100Cr6-std</td>
<td>Standard (austenitising ~860°C, bainitising 250 °C)</td>
<td>Conventional lower bainite + coarse globular carbides</td>
</tr>
<tr>
<td></td>
<td>100Cr6-950C-250C</td>
<td>Austenitising 950 °C, bainitising 250 °C</td>
<td>Conventional lower bainite</td>
</tr>
<tr>
<td></td>
<td>100Cr6-950C-300C</td>
<td>Austenitising 950 °C, bainitising 300 °C</td>
<td>Conventional lower bainite</td>
</tr>
<tr>
<td>42CrMo4</td>
<td>42CrMo4-B</td>
<td>Austenitising 860°C, bainitising at 350 °C</td>
<td>Bainite</td>
</tr>
<tr>
<td></td>
<td>42CrMo4-B</td>
<td>Austenitising 860°C, bainitising at 400 °C</td>
<td>Bainite</td>
</tr>
<tr>
<td></td>
<td>42CrMo4-QT</td>
<td>Quenched and tempered</td>
<td>Tempered martensite</td>
</tr>
</tbody>
</table>

Table 21: Additional grades and heat-treatments added as references for comparison of wear resistance.

5.2.2 Results

As shown in Figure 24, the alloys designed in the present project exhibit wear resistance significantly superior to that of standard material of similar hardness (100Cr6 for example). It is also worth noting that the results achieved on industrial material (1CSi-ind and 06CV) show again significant improvement against similar laboratory casts of the same grades. It is also worth underlining that SWR on 1CSi-indus-220C or 06CV-220C were about 1% or less of that measured of 100Cr6 bainitised to similar hardness.

Figure 24: Specific wear rate (SWR) for all the conditions investigated in the present project. The newly designed grades exhibit significantly lower SWR when compared to conventional bainitic or quenched and tempered microstructures of similar hardness.
5.2.3 Analysis, role of microstructural parameters

Hardness changes
Analyses carried out on worn surfaces and cross-sections are detailed in Appendix F. No significant differences were observed in terms of damage mechanisms.

Hardness profiles underneath the contact surface were investigated in all specimens (Appendix F). While no correlation could be identified between the hardened layer depth and the performance in rolling/sliding wear, the maximum hardness achieved on the surface was shown to have a significant influence (Figure 25).

![Figure 25: Specific wear rate as a function of maximum hardness measured underneath the contact surface.](image)

Retained austenite
Retained austenite measurements under the contact surface were carried out by ASCOMETAL. Electropolishing was used to remove, first, 25 µm of material, then 200 µm. X-Ray measurements of retained austenite were then carried out on the etched surfaces. As shown in Table 22, the main difference between Nanobain grades and standard 100Cr6 is the presence of retained austenite. The latter is destabilised during wear tests as shown by the reduction of its fraction in the vicinity of the surface.

<table>
<thead>
<tr>
<th>Reference</th>
<th>R. A. / %</th>
<th>Surface hardness after testing</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>@ 25 µm</td>
<td>@ 200µm</td>
</tr>
<tr>
<td>1CSi-indus-220C</td>
<td>29</td>
<td>30</td>
</tr>
<tr>
<td>06CV-220C</td>
<td>16</td>
<td>32</td>
</tr>
<tr>
<td>06C-250C</td>
<td>7</td>
<td>17</td>
</tr>
<tr>
<td>100Cr6-300C</td>
<td>3,7</td>
<td></td>
</tr>
</tbody>
</table>

Table 22: Retained austenite content 25 and 200 µm underneath the contact surface of some rolling/sliding specimens, and surface hardness after testing.

The case of 1CSi-indus-220C is particularly interesting. This grade achieved the best wear performance measured in the project, with a specific wear rate less than 1% that of 100Cr6 bainitised to a similar hardness. Measurements of retained austenite content indicated absence of destabilisation. However, when comparing hardness profiles between, for example, 1CSi-indus-220C and 100Cr6-950C-300C (Figure 26), a difference was clearly highlighted in that the hardness increase in 1CSi-indus is strongly localised. Indeed, at 25 µm under the surface, the measured hardness was virtually identical to the bulk one.
It is therefore not clear whether the hardness increase achieved in 1CSi is the result of retained austenite decomposition within the first 20µm or less under the surface, or the result of a more conventional form of work-hardening. The case of 100Cr6, which contains negligible amounts of retained austenite, indeed demonstrates that at least some fraction of the hardness increase occurs without the need for the decomposition of this phase.

![Figure 26](attachment:Figure_26.png)

Figure 26: Hardness profiles under the worn surface for 1CSi-indus-220C and 100Cr6-950C-300C. The 1CSi exhibits hardening limited to 15 µm or less.

Nevertheless, the results, in general, demonstrate the beneficial role of retained austenite in all Nanobain grades, and may explain why these make up a separate family on the wear vs. hardness graph (Figure 24).

Cleanliness

Within the set of Nanobain grades, it is clear that industrial heats achieved much better results in rolling/sliding wear than laboratory casts. This may be explained if one considers the damage mechanism at hand. As detailed in Appendix F, examinations of tested specimens are consistent with damage mainly occurring through a rolling contact fatigue mechanism (RCF). It is well established that cleanliness is an important factor in controlling the performance of high strength steels under RCF. The inclusion content was thus examined in two alloys heat-treated to a similar hardness: 06C-250C and 06CV-220C, the latter being an industrial cast. Details of the measurements are given in Appendix F. Results clearly indicate that both oxides and sulphides content and sizes are significantly higher for the laboratory heat (as expected from measured S and O content and from manufacturing routes). Indeed, oxide alignments near 1 mm in length were found in 06C-250C, the 06CV grade exhibiting cleanliness similar to that achieved on industrial 1 %C bearing steels such as 100Cr6. It is thus not unlikely that the additional improvement achieved on industrial casts is related to improvement in cleanliness.

It is therefore also remarkable that laboratory heats with relatively poor cleanliness performed significantly better than industrial 100Cr6 manufactured to bearing cleanliness standard. This suggests once again that retained austenite content and stability is a factor of primary importance in explaining the rolling/sliding wear performance of Nanobain grades.

Influence of globular carbides in 100Cr6

The results achieved are also the opportunity to discuss the influence of globular carbides in 100Cr6. As explained earlier, the reference condition provided by Bosch to LTU is 100Cr6 austenitised at a temperature below Acm, so that an important fraction of the globular carbides resulting from earlier spheroidising are kept part of the microstructure. As shown in Figure 24, rolling sliding SWR results were identical for 100Cr6, whether austenitisation had been carried out at 860 °C (globular carbides) or at 950 °C (all carbon in solution).
5.3 TASK 5.2 AND 5.3: HIGH-PRESSURE ABRASION TESTS, INFLUENCE OF MICROSTRUCTURE

5.3.1 Introduction

It was initially intended to carry out ball on block impact tests as part of Task 5.3; however, the quantity of material available from laboratory casts (20 or 35 kg) turned out to be largely insufficient for this particular test. Therefore, V-notched Charpy impact tests were used instead.

5.3.2 High pressure abrasion and impact testing

Details of the testing method are presented in Appendix G. In addition to the references already described in Table 20, METSO also made use of its internal reference materials LBG2 and Hardox 500. The former is a medium-carbon tool steel with high Cr content, quenched and double tempered to a hardness of 500 HV. The latter is an abrasion resistant steel typically containing (for the range of thickness of interest): 1.5% Cr, 1.5% Ni, 0.6% Mo and heat-treated to a hardness of approx. 500HV.

The final results of the wear tests are shown in Figure 27 below. Variations in the abrasion resistance between different compositions and heat treatments were noticed and conclusions for optimal material selection can be made based on the results.

![Figure 27: High pressure abrasive wear test results. References are presented in Table 21. LBG2 and Hardox are additional references from METSO.](image)

![Figure 28: Impact toughness results.](image)
The best possible abrasion resistance is not the only requirement for optimal material selection but the impact toughness also plays a very important role. Impact tests on Charpy V-notch specimens were performed on 7 variants. Tests were performed at room temperature according to standard, with an impact force of 300 N. Impact tests results are shown in Figure 28.

The highest impact toughness for the designed alloys was achieved with sample 06CNb-250. On the 08C grade, the influence on bainite transformation temperature was investigated. Increasing transformation temperature also increases the impact toughness; which is in good correlation with the mechanical properties. Also the influence of alloying elements was studied. For the 06C concept Nb addition very much increases the toughness. As well as for the 1C concept the addition of Si increases the toughness properties.

![Figure 29: The combination of wear and toughness properties.](image)

The tests carried out at METSO lead to a ranking of the different grades and transformation conditions used. The best high-stress abrasion resistance values were achieved by 08C-270, 08C-250 and 06C-250, the best impact toughness values by 06CNb-250, 1CSi-250 and 08C-270, and the best combination of those two properties lead to the following ranking:

1) 08C-270
2) 06CNb-250
3) 1CSi-250

Since the 08C grade was not, however, designed to offer the hardenability required for METSO’s component, and since major modifications would have to be made to address this issue, it was decided that industrial tests would be carried out using a material similar to 06CNb.

5.4 CONCLUSIONS AND ACHIEVEMENTS

5.4.1 Significant deviations from work plan

As mentioned earlier, the material requirement for the ball-on-block tests initially planned is such that it was not possible to carry out Task 5.3. As a substitute, impact tests were used. There were also significant delays in Task 5.1, with a full set of results only available at the end of the project instead of mid-term. Delays were party due to small delays carried over from previous tasks (2-3 months), and mainly to unavailability of equipment.

5.4.2 Main results and conclusions

Whether discussing the rolling/sliding or high pressure abrasive wear tests, the performance of Nanobain material has been found to be on par with significantly more expensive alloys such as those in...
use at Metso, or largely improved compared to Bosch's reference (bainitised 100Cr6). Indeed, in rolling-sliding wear tests, industrial materials (1CSi and 06CV) exhibited specific wear rates as little as 1% of that measured on 100Cr6 heat-treated at the same hardness.

Performance in rolling-sliding tests appeared to be significantly improved by the presence of significant quantities of retained austenite in the Nanobain alloys as opposed to conventional bainite microstructures. The decomposition of this retained austenite could explain the surface hardness measured on Nanobain grades, which were significantly higher than that measured on conventional bainitic 100Cr6. In turn, it is shown that surface hardness and wear rate are directly related.

Cleanliness may also explain the differences observed between laboratory and industrial casts of Nanobain material. However, that better results were achieved on laboratory casts with poor cleanliness and reduction ratio (typically 8), when compared to industrial material with bearing steel standard cleanliness and high reduction ratio (typically > 50), indicates that this remains a factor of secondary importance.

Finally, tests carried out at METSO lead to the selection of a candidate material for manufacture of industrial components.

6. **WP6 : TENSILE TEST AND OF NANOBAINITIC STEELS**

6.1 **OBJECTIVES AND INTRODUCTION**

WP6 had two objectives:
- to establish the tensile and fatigue performance of Nanobain steels
- to identify relevant microstructural parameters
- to establish a relative value of the contact fatigue resistance of carbide free and carbide containing microstructures

6.2 **TASK 6.1 : TENSILE AND IMPACT PROPERTIES**

6.2.1 **Overview**

Tensile test were performed on 16 different variants, using tensile specimens with maximum diameter 12 or 15 mm (heads). Table 23 shows an overview of all tested samples and their chemical composition and heat treatment.

<table>
<thead>
<tr>
<th>Name</th>
<th>Steel</th>
<th>Austenisation</th>
<th>Bainitizing</th>
<th>Done by</th>
</tr>
</thead>
<tbody>
<tr>
<td>06C-250</td>
<td>06C</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>06CCr-250</td>
<td>06CCr</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>06CMo-250</td>
<td>06CMo</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>06CNb-220</td>
<td>06CNb</td>
<td>1h@890°C</td>
<td>22h@220°C</td>
<td>ALD</td>
</tr>
<tr>
<td>06CNb-250</td>
<td>06CNb</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>08C-220</td>
<td>08C</td>
<td>1h@890°C</td>
<td>22h@220°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>08C-250</td>
<td>08C</td>
<td>1h@890°C</td>
<td>16h@250°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>08C-270</td>
<td>08C</td>
<td>1h@890°C</td>
<td>7h@270°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1C-250</td>
<td>1C</td>
<td>1h@950°C</td>
<td>16h@250°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CMo-200</td>
<td>1CMo</td>
<td>1h@950°C</td>
<td>64h@200°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CMo-220</td>
<td>1CMo</td>
<td>1h@950°C</td>
<td>22h@220°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CMo-250</td>
<td>1CMo</td>
<td>1h@950°C</td>
<td>16h@250°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CNb-220</td>
<td>1CNb</td>
<td>1h@950°C</td>
<td>22h@220°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CNb-240</td>
<td>1CNb</td>
<td>1h@950°C</td>
<td>16h@240°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CSi-220</td>
<td>1CSi</td>
<td>1h@950°C</td>
<td>22h@220°C</td>
<td>Ascometal</td>
</tr>
<tr>
<td>1CSi-250</td>
<td>1CSi</td>
<td>1h@950°C</td>
<td>16h@250°C</td>
<td>Ascometal</td>
</tr>
</tbody>
</table>

Table 23 : Overview of all tested tensile variants and their heat treatment.
Tests were performed at room temperature according to standard with constant strain rate of 0.004 s\(^{-1}\). Figure 30 shows an overview of all results on tensile testing. Results are the average value of at least three samples except for 1CNb-220 (two samples) and 1CMo-220 (one sample).

These results show that all samples reached a UTS of at least 2000 MPa, with uniform elongation reaching in some cases 10%. Note that all results are given as engineering strain / stress.

![Graph showing tensile test results](image)

**Figure 30**: Overview of tensile test results.

### 6.2.2 Effect of alloying content and temperature

Figure 31 shows the influence of alloying element on all grades bainitized at identical temperature (250 °C). This comparison clearly outlines three groups: 06Cx grades do not exhibit significant difference in tensile properties as a function of composition. For a same transformation temperature however, higher carbon grades (08C, having actually 0.9%C, 1C and 1CMo) exhibit higher tensile
strengths (typically > 2200 MPa) and lower elongations. Finally the 1CSi grade seems an exception with a high tensile strength and the highest uniform elongation (> 10%).

![Tensile properties of all grades tested after transformation at 250 °C.](image)

This trend is partially confirmed when considering samples transformed at 220 °C (Figure 32): the 06CNb grade exhibits lower YS than higher carbon grade although in this case, the UTS is similar. The reason for this is unclear: it was noted that on all five 06CNb-220°C tensile specimens, the tensile curve exhibits a much larger work hardening than for transformation at 250 °C. Comparison of the results obtained on identical grades at different temperatures typically outlines the expected behavior; that is, an increase in tensile properties and decrease in ductility for lower transformation temperatures.
6.2.3 Conclusions

Tensile test results have demonstrated that target properties were achieved if not exceeded in many cases. UTS can be in excess of 2300 MPa and, in the case of the 1CSi alloy, a true uniform elongation of 17% has been reached, for a UTS of over 2000 MPa. These values are considerably above those exhibited by bainitised standard 100Cr6.

Comparison of results obtained on different grades do not allow identification of any influence of substitutional alloying elements (Cr, Mo, Si, etc), but reveal significant differences between 06Cx and 1Cx grades.

6.3 Task 6.2: Fatigue Resistance

6.3.1 Introduction

It was initially expected that task 6.3 could be completed before a selection had to be made as to the compositions of the industrial heats. However, because of the need to cast additional material for the sole purpose of fatigue testing, this appeared not to be possible. In order to retain the possibility to test industrial material, it was thus decided to base the material selection on task 6.1 and carry out rotation bending fatigue testing in parallel.

6.3.2 Rotation bending fatigue testing

Following initial tests on 1CNb grade the distribution of tests was modified to obtain reliable fatigue data not influenced by the poor cleanliness of lab casts. Results are detailed in Appendix M, and a summary is shown in Table 24.
Table 24 : 10^7 cycles fatigue strength as determined on notched rotation bending specimens.

<table>
<thead>
<tr>
<th>Condition</th>
<th>UTS / MPa</th>
<th>Sa, 50% at 10^7 cycles / MPa for R = -1, Kt = 1.6 by rotation bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CNb-220-FAT</td>
<td>2073</td>
<td>430</td>
</tr>
<tr>
<td>06C-250-FAT</td>
<td>2023</td>
<td>665</td>
</tr>
<tr>
<td>08C-270-FAT</td>
<td>2036</td>
<td>440</td>
</tr>
<tr>
<td>1CSi-ind-220-FAT</td>
<td>2224</td>
<td>550</td>
</tr>
<tr>
<td>1CSi-ind-250-FAT</td>
<td>2072</td>
<td>535</td>
</tr>
<tr>
<td>06CV-ind-250-FAT</td>
<td>2003</td>
<td>690</td>
</tr>
<tr>
<td>06CV-ind-270-FAT</td>
<td>1822</td>
<td>675</td>
</tr>
</tbody>
</table>

In the case of 1CSI, as detailed in Appendix M, all of the failures had initiated on inclusions. This was not so clearly the case in 1CNb, although the latter exhibited a lower fatigue strength. It is not unlikely that porosity was present in the laboratory heats, which may have initiated early failures.

The only clearly identified trend in the results shown in Table 24, is that hypoeutectoid steels (06C-250C, 06CV-250C and -270C) all exhibited significantly higher fatigue strength than higher carbon grades (080C and 1Cx). As will be discussed later, this was also the case during pulsating tests carried out on component demonstrator.

The fatigue strength of both 06C (laboratory heat) and 06CV (industrial heat) were found to be similar, in spite of significant differences in cleanliness (see end of Appendix F). This is in agreement with the absence of identified initiations on inclusions in these specimens.

6.3.3 Standing contact fatigue testing

During the first half of the project, LTU investigated the possibility to carry out standing contact fatigue tests on grades developed within the project. These are detailed in Appendix N. Number of cycles to failure was found to decrease rapidly with hardness and the method was not thought to be adapted for such high strength material.

6.4 Task 6.3: Influence of microstructure on tensile and fatigue properties

The objective of task 6.3 was to correlate tensile and fatigue results with microstructural parameters. As will be shown, tensile properties can reasonably well be explained through the parameters investigated. However, ductility, toughness and fatigue performance could not be easily explained. Indeed, specimens with apparently identical microstructures (same retained austenite, lath thickness) exhibited significantly different tensile ductility and toughness.

In carbide free bainitic microstructures, strength is expected to depend primarily on retained austenite content, with the yield strength decreasing with increasing fraction of retained austenite. The bainitic microstructure strength is itself primarily a function of the lath thickness. For the morphologies at hand, the dependency between yield strength and lath thickness is expected to be in \( t_{\beta}^{-1} \).

Thus, strength should increase with decreasing retained austenite fraction (i.e. increasing fraction of bainite \( V_{\beta}=(1-V_\gamma) \), and with \( t_{\beta}^{-1} \). \( V_\gamma \) is the volume fraction of retained austenite. As shown in Figure 33, there is indeed a good correlation between yield strength and \( V_{\beta}/t_{\beta} \).
The question of tensile ductility and toughness is much more difficult to address. As shown in Table 25, a number of apparently similar microstructures (tβ, Vγ) led to very different properties.

<table>
<thead>
<tr>
<th></th>
<th>UE</th>
<th>TE</th>
<th>C</th>
<th>Vγ</th>
<th>RArea</th>
<th>KV</th>
<th>tβ</th>
<th>HV</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>08C</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>220</td>
<td>3.2</td>
<td>4.1</td>
<td>1.05</td>
<td>22</td>
<td>6.4</td>
<td>5</td>
<td>32</td>
<td>710</td>
</tr>
<tr>
<td>250</td>
<td>3.1</td>
<td>12</td>
<td>1.41</td>
<td>18</td>
<td>31.7</td>
<td>10</td>
<td>38</td>
<td>659</td>
</tr>
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<td>270</td>
<td>4.5</td>
<td>12.7</td>
<td>1.47</td>
<td>24</td>
<td>25.2</td>
<td>13</td>
<td>36</td>
<td>615</td>
</tr>
<tr>
<td><strong>1CSI</strong></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>220</td>
<td>7.2</td>
<td>7.2</td>
<td>1.2</td>
<td>36</td>
<td>9</td>
<td>28</td>
<td>650</td>
<td></td>
</tr>
<tr>
<td>250</td>
<td>11.6</td>
<td>21.3</td>
<td>1.5</td>
<td>34</td>
<td>32.4</td>
<td>15</td>
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<td>625</td>
</tr>
<tr>
<td><strong>06CV</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>220</td>
<td>6.7</td>
<td>6.7</td>
<td>24</td>
<td>9.3</td>
<td>5</td>
<td>38</td>
<td>643</td>
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<td>250</td>
<td>8.81</td>
<td>11.9</td>
<td>22</td>
<td>17.6</td>
<td>17</td>
<td>37</td>
<td>600</td>
<td></td>
</tr>
<tr>
<td>280</td>
<td>12.4</td>
<td>17.8</td>
<td>31</td>
<td>20.1</td>
<td>24</td>
<td>48</td>
<td>554</td>
<td></td>
</tr>
</tbody>
</table>

Table 25: Uniform elongation (UE), total elongation (TE), estimated carbon content in the retained austenite (C), retained austenite content, reduction of area, V-notch impact toughness, bainite lath thickness and hardness of a number of conditions investigated in the project.
As an example, 1CSi exhibits identical retained austenite and bainite lath thickness after heat-treatment at 220 and 250 °C, but its tensile ductility is entirely different. Similarly, the 08C and 06CV grades do not exhibit significant microstructural changes between 220 and 250 °C, though the toughness and tensile ductility are considerably improved at 250 °C.

Appendix O examines the different mechanisms that may explain the presence of considerable ductility in some of the specimens tested. As detailed in the appendix, it was possible to exclude a contribution of twin induced plasticity in the retained austenite. Nevertheless, a clear correlation was found between retained austenite content and deformation, the former decreasing progressively with increasing deformation up to a minimum of approximately 10%. The analysis suggests that retained austenite morphology and stability (influenced amongst other parameters, by its carbon content) plays a major role in explaining the differences in ductility identified between the different specimens. This should be confirmed with appropriate investigation techniques such as 3D atom probe tomography, techniques which were not available within the scope of this project.

As discussed earlier, fatigue performances are significantly more difficult to relate to the microstructural parameters investigated within the scope of the project. Whilst cleanliness certainly had an influence, satisfactory results were achieved on all lower carbon grades (Table 24) regardless of their manufacturing route. With results on par with the current bainitised reference at Bosch (100Cr6), it is hoped that a full understanding of the fatigue resistance of carbide free, nano-structured bainite will lead to the development of a grade of superior performances.

6.5 CONCLUSIONS AND ACHIEVEMENTS

6.5.1 Significant deviations from work plan

Significant delays were experienced in the rotation bending fatigue tests, mostly due to the need for additional material. The choice of grades to be manufactured industrially was thus based on tensile results and partial fatigue results. The rotation bending fatigue test programme was carried out in parallel with the characterization of the industrial heats, which were included in the testing material. This had the advantage of providing one set of test results including both laboratory and industrial casts.

6.5.2 Main results and conclusions

The fatigue strength of various nanostructured carbide-free bainitic steels has been investigated. Results achieved on some of the grades (06CV and 06C) were on par with those obtained on bainitised 100Cr6 in spite of a lower UTS. Results achieved on higher carbon grades were systematically worse, though explanation could be found in spite of detailed microstructural investigations.

Results achieved in terms of tensile properties were exceptional and somewhat unexpected. A yet unreported combination of UTS above 2 GPa and uniform elongation above 10% was obtained on the bainitised 1CSi material. Yield strength and hardness were found to correlate well with the microstructural parameters investigated, i.e. bainite lath thickness and retained austenite content. However, ductility and impact toughness could not be explained. In fact, specimens with apparently similar microstructures (lath thickness, retained austenite) gave significantly different results. In particular, bainitising at 250 °C instead of 220 °C had a non detectable effect on the microstructure but lead to a pronounced improvement in toughness and ductility. This is thought to be related to retained austenite stability rather than content. Further experimental evidence should be sought using adapted equipment.
7. WP7: INDUSTRIAL HEATS

7.1 OBJECTIVES

WP7 had three objectives:
- to provide industrial heats for component testing
- to validate the transformation behaviour and mechanical properties at the industrial scale
- to assess the application potentialities of this new class of steels

7.2 TASK 7.1: MANUFACTURING OF INDUSTRIAL MATERIAL

7.2.1 Introduction

Task 7.1 is concerned with the manufacturing of industrial material for both target applications (UTS > 2000 MPa for Bosch, emphasis on fatigue resistance, UTS > 1600 MPa for METSO, emphasis on wear resistance).

7.2.2 0.6%C base industrial grade (06CV)

Prior to manufacturing a 100T cast, and due to the significant changes between the target composition for the industrial alloy and the experimental grades, it was decided to manufacture one additional laboratory heat and confirm both the critical cooling rate for ferrite-pearlite formation and the absence of significant issues with hot-ductility. This work is reported in Appendix H. From these results, it was confirmed that the hardenability was exactly that required for the component, and that hot ductility was sufficient for hot-rolling though care had to be taken with the reheating temperature.

A 100T industrial heat was manufactured at Ascometal's Fos plant in July 2010. It was cast as 7.2T ingots, and hot-rolled to 480x480 blooms. These were later reheated and hot-rolled to a diameter of 300 mm, which were shipped to METSO after tempering 6 h at 680 C. The entire process went without particular difficulties. The microstructure was pearlite, with a hardness of approximately 320 HV (30kg load). Details of the composition are provided in Table 26. In addition, an oxygen content of 4 ppm was achieved (see Appendix E, Table 39 for details).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Al</th>
<th>Ti</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>625</td>
<td>1580</td>
<td>1230</td>
<td>0</td>
<td>0</td>
<td>180</td>
<td>1700</td>
<td>145</td>
<td>100</td>
<td>20</td>
<td>0</td>
</tr>
<tr>
<td>Max</td>
<td>675</td>
<td>1700</td>
<td>1350</td>
<td>10</td>
<td>10</td>
<td>250</td>
<td>1800</td>
<td>165</td>
<td>300</td>
<td>35</td>
<td>3</td>
</tr>
<tr>
<td>Cast 60840</td>
<td>673</td>
<td>1667</td>
<td>1315</td>
<td>2</td>
<td>8</td>
<td>202</td>
<td>1730</td>
<td>149</td>
<td>180</td>
<td>30</td>
<td>3</td>
</tr>
</tbody>
</table>

Table 26: Target and achieved compositions for the 06C industrial grade.

To allow testing by BOSCH, additional material was hot-rolled to smaller blooms and shipped to Ascometal's Hagondange plant, where it was hot-rolled to a diameter of 65 mm. Contrary to expectations however, the 06C industrial grade was found to be air hardenable in bars of this diameter and natural cooling led to dramatic cracking of all bars (hardness ~700HV). A second hot-rolling was therefore carried out at Fos plant to a diameter of 120 mm. Bars were tempered in similar conditions as for the 300 mm diameter bars. The resulting microstructure was similar (300HV, pearlite).

7.2.3 1%C base industrial grade (1CSi-indus)

The industrial heat No.17817, steel grade 100SiCr10-4, was manufactured in Gerdau Sidenor Reinosa plant by ingot casting. The composition is given in Table 27 (O₂ content was 18 ppm). In comparison with the 1CSi laboratory cast, Cr has been increased to enhance hardenability, and Si decreased to reduce manufacturing difficulties.

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>V</th>
<th>Cu</th>
<th>Al</th>
<th>Ti</th>
<th>Ca</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>on tundish</td>
<td>0.99</td>
<td>0.75</td>
<td>2.45</td>
<td>0.008</td>
<td>0.002</td>
<td>0.98</td>
<td>0.12</td>
<td>0.024</td>
<td>0.002</td>
<td>0.19</td>
<td>0.024</td>
<td>0.007</td>
<td>0.0016</td>
</tr>
<tr>
<td>on final product</td>
<td>0.99</td>
<td>0.74</td>
<td>2.47</td>
<td>0.008</td>
<td>0.003</td>
<td>0.97</td>
<td>0.12</td>
<td>0.028</td>
<td>0.003</td>
<td>0.17</td>
<td>0.024</td>
<td>0.008</td>
<td>0.0013</td>
</tr>
</tbody>
</table>

Table 27: Composition of industrial heat 17817 manufactured by Sidenor for the 1CSi industrial grade.
To hot roll the ingots, hot ductility data from workability tests of 1CSi experimental heat were used (Figure 35). Therefore, a temperature of 1100ºC-1180ºC was specified for reheating in pit furnaces.

Square billets of 160x160 mm² were hot rolled and, grinded to remove surface defects. For components fatigue tests, hot rolling into 35-mm bars was defined. Hot rolling was through drag-over mill, continuous mill and finishing Kocks. The microstructure in as rolled condition was mostly pearlite, showing poor ductility and toughness (Table 28).

<table>
<thead>
<tr>
<th>Rm (Mpa)</th>
<th>Re (Mpa)</th>
<th>A-5d (%)</th>
<th>Z (%)</th>
<th>KU (20ºC) (J)</th>
<th>HV30</th>
</tr>
</thead>
<tbody>
<tr>
<td>1182,8</td>
<td>831,6</td>
<td>3,4</td>
<td>0</td>
<td>3</td>
<td>390</td>
</tr>
</tbody>
</table>

Table 28 : Mechanical properties of a 35 mm diameter 1CSi bar in as-rolled condition.

In order to make easier handling, machining and further operations, subcritical annealing at 700ºC, 8 hours were performed, which resulted in the mechanical properties detailed in Table 29.

<table>
<thead>
<tr>
<th>Rm (Mpa)</th>
<th>Re (Mpa)</th>
<th>A-5d (%)</th>
<th>Z (%)</th>
<th>KU (20ºC) (J)</th>
<th>HB</th>
</tr>
</thead>
<tbody>
<tr>
<td>1252,2</td>
<td>697</td>
<td>9</td>
<td>14,6</td>
<td>2,7</td>
<td>320</td>
</tr>
</tbody>
</table>

Table 29 : Mechanical properties of a 35 mm diameter 1CSi bar in as-delivered condition (tempered).

35 mm bars were supplied to LTU, BOSCH and CENIM.

7.2.4 Conclusions

Both industrial heats lead to a number of difficulties during manufacturing. Those encountered on the 06CV were mostly related to the high hardenability of the material. These difficulties imply that hot-rolling to small diameter or manufacture of small size components will require special care to avoid air hardening, but in all cases can be addressed successfully.

In contrast, difficulties with the 1CSi grade included not only the risk of air hardening for small components, but mostly issues with surface quality, extremely poor ductility in the as-rolled condition, etc. It is not clear, at this stage, how easily such difficulties can be overcome to render the alloy economically viable.

7.3 Task 7.2 : Characterisation of industrial materials

7.3.1 Introduction

The aim of task 7.2 was to characterize both industrial alloys through laboratory testing. Thus, tensile testing after bainitising, rotation bending fatigue and wear tests were carried out. Results of wear tests are discussed as part of WP5 (section 5), while rotation bending fatigue tests are included with those on laboratory heats in WP6 (section 6).

7.3.2 Tensile tests

A first testing campaign had been carried out by Sidenor on the 1CSi grade. However, results were significantly different from those achieved on laboratory casts and it was suspected that temperature
control during the (sub-contracted) salt-bath heat-treatment was not satisfactory. Results are given in Appendix I. Tensile tests and corresponding heat-treatments (220 °C, 250 °C and 270 °C) were then carried out by Bosch on both 1CSi-indus and 06CV materials. Results are shown in comparison with the corresponding lab casts in Figure 36 for 06CV and in Figure 37 for 1CSi.

![Figure 36: Tensile properties of industrial cast 06CV at three different bainitising temperatures (220°C, 250°C and 280°C) in comparison with the lab cast 06C bainitized at 250°C.](image)

As shown in Figure 36 and Figure 37, yield and tensile properties strength were similar if not identical to those achieved on laboratory heats. Uniform elongation was also similar to results achieved on laboratory casts, and the exceptional combination of strength and ductility (above 10% uniform elongation for 2GPa) was once again achieved with 1CSi heat-treated at 250 or 270 °C.

7.3.3 Wear performances and impact toughness

Results of rolling-sliding wear tests are discussed in section 5. It is only reminded here that specific wear rates achieved on both industrial heats were as little as 1% of that measured on bearing steel quality 100Cr6 heat-treated to a similar hardness. As discussed in WP5, it is thought that the combination of significant amounts of retained austenite (typically 30% in the tested conditions) and improved cleanliness led to those results.

METSO carried out additional high pressure abrasion tests (see 5.3.2) on material issued from industrial 06CV. As previously, results are shown against those achieved on a number of internal references for METSO.
Relative wear resistance (ref: Hardox 500)

Figure 38: Relative wear resistance for 06CV heat-treated at three different temperatures, against previously discussed results.

Because impact toughness is critical for the applications to be tested by METSO, V-notched impact tests were carried out on 06CV bainitised at three temperatures. The results are presented in Table 30. The results of metallographic examinations carried out on these samples are also reported. Because of a concern that toughness would be insufficient to allow safe operation, it was decided to heat-treat the industrial components at 280 °C.

<table>
<thead>
<tr>
<th></th>
<th>KV / J</th>
<th>Hardness / HV30</th>
<th>Lath thickness / nm</th>
<th>Retained austenite / %</th>
</tr>
</thead>
<tbody>
<tr>
<td>06CV-220C</td>
<td>5</td>
<td>628</td>
<td>38</td>
<td>24</td>
</tr>
<tr>
<td>06CV-250C</td>
<td>17</td>
<td>600</td>
<td>37</td>
<td>22</td>
</tr>
<tr>
<td>06CV-280C</td>
<td>24</td>
<td>555</td>
<td>48</td>
<td>31</td>
</tr>
</tbody>
</table>

Table 30: V-notch impact toughness and hardness of heat-treated specimens of 06CV. Also indicated are bainite lath thickness and retained austenite measured on these samples.

7.3.4 Tension-tension fatigue performance on notched laboratory specimens

As explained in an earlier section, fatigue performances on rotation bending were ultimately carried out on some of the industrial material. This had the benefit of allowing comparison between laboratory and industrial materials in identical testing conditions. Results obtained on rotation bending specimens are discussed in section 6.

Fatigue testing on the industrial heats was carried out using two methods:
- tensile-tensile tests on notched specimens
- internal pulsating tests on "component demonstrators"

This part is primarily concerned with notched tensile-tensile fatigue tests. The results achieved on component demonstrators will be discussed as part of Task 7.3.

Details of testing procedures and raw results are presented in Appendix J. Results are summarised in Table 31. For comparison purposes, the fatigue strength as measured in similar conditions for 100Cr6 and 50CrMo4 has been indicated. Both are reference materials for the manufacturing of diesel injection components. The results clearly show that, in spite of its significantly lower UTS than 100Cr6, the 06CV industrial grade exhibited nearly identical performances. It must also be reminded that, with most
failures initiated on Ti(C,N) in the 06CV grade, there is immediate potential for improvement since the concerned manufacturing plant routinely achieves Ti contents of 10 ppm and lower in bearing steels.

<table>
<thead>
<tr>
<th></th>
<th>06CV-250</th>
<th>06CV-270</th>
<th>1CSI-220</th>
<th>1CSI-250</th>
<th>100Cr6 [TL], 1998</th>
<th>50CrMo4 [SSc], 2012</th>
</tr>
</thead>
<tbody>
<tr>
<td>UTS / MPa</td>
<td>2003</td>
<td>1822</td>
<td>2224</td>
<td>2072</td>
<td>2350</td>
<td>1180</td>
</tr>
<tr>
<td>$S_{a,50%}$ at $N = 10^7$ cycles / MPa</td>
<td>350</td>
<td>330</td>
<td>205</td>
<td>245</td>
<td>355</td>
<td>305</td>
</tr>
</tbody>
</table>

Table 31: Comparison of the fatigue strength at $10^7$ cycles of nanobainitic steels 06CV and 1CSI with steels nowadays used in diesel injection systems such as 100Cr6 and 50CrMo4 tested in tension-tension (R = 0,1) and notched conditions ($K_t = 2$).

The relatively low fatigue strength of 1CSI appears to be related to the occurrence of cleavage fracture areas, which are not usually observed in such fatigue tests. These were initially thought to be related to exaggerated grain growth during austenitising. However, measurements carried out at CENIM using thermal etching have shown the prior austenite grain size to be approximately 14 µm in similar conditions as those used for austenitising.

### 7.4 TASK 7.3 AND 7.4: COMPONENT MANUFACTURE, CHARACTERISATION AND TESTING

#### 7.4.1 Introduction

The aim of Task 7.3 was to manufacture component or component demonstrators from industrial material and test them in situ or in representative conditions. Task 7.4 was concerned with the metallographic examination of the thus manufactured specimens.

#### 7.4.2 Manufacture, testing and examination of METSO's component

It was eventually decided that METSO would manufacture scrap shear blades from the 06CV material, as field testing of crusher components would not be achieved within the imparted time for the project.

From the 300 mm bars, METSO forged 280x 75x2000 and 210x85x1800 mm bars, using furnace cooling to avoid cracking. These bars were the starting point for manufacturing of field test components which were then heat-treated in salt bath (austenitising 890 °C, bainitising 280 °C for 7 h).

Sections of one component were sent to ASCOMETAL for metallographic examination. Details are provided in Appendix K. The Vickers hardness (540 HV) was found to be in good agreement from what was expected of the heat-treatment temperature, and remarkably homogeneous given the size of the component: over a total of 32 measurements spreading from the core of the component to its corner, hardness values did not fall below 533 or exceed 549 HV. As indicated in Appendix K, bainite lath thickness was measured to 58 nm, in agreement with earlier measurements.

Tensile and Charpy impact toughness tests specimens were manufactured from METSO's component and tested at ASCOMETAL. Results are shown in Table 32.

<table>
<thead>
<tr>
<th>YS / Mpa</th>
<th>UTS / Mpa</th>
<th>U. El. / %</th>
<th>T. E. / %</th>
<th>KCV / J.cm$^{-2}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1233</td>
<td>1717</td>
<td>8.4</td>
<td>10.3</td>
<td>22</td>
</tr>
</tbody>
</table>

Table 32: Results from tensile and Charpy impact toughness tests carried out on specimens machined from the component manufactured and heat-treated by METSO.

Thus, metallographic examination confirmed the successful design of the alloy, since mechanical properties did not change significantly between laboratory specimens and large components, as expected from tailoring of the hardenability.
7.4.3 Field testing of METSO's component

As detailed in Appendix L, the 06CV component manufactured by METSO was a scrap shear blade. This was tested at the Karjaa site, Finland. The standard material for this application is 45NiCrMoV16-6 (1.2746), a 1.5Cr-4Ni-0.8Mo-0.5V with which blades normally provide 8-12000 cuts over their lifetime.

The number of cuts achieved with the 06CV blade was lower though of similar order of magnitude. The reduction is thought to be partly due to changes in the feeding sequence, with very damaging rail steel having been introduced earlier than usual.

Overall, it is felt by METSO that wear performance should undoubtedly be on par with the standard material once some fine tuning carried out, and for similar testing conditions. The cost benefit is estimated to 10-20% due to the significantly cheaper material, though slightly offset by the heat-treatment duration.

7.4.4 Manufacturing and testing of diesel injection component demonstrators

To evaluate the potential for industrial application in high loaded diesel injection systems, investigations using so-called technological demonstrators were carried out. The geometry of the component demonstrator is shown in Figure 39. The specimen used for these tests have the same characteristic bore intersection as a rail body.

![Geometry of the component demonstrator manufacture by BOSCH.](image)

The manufacturing process of both specimens is soft machining, salt bath heat treatment and hard machining. Internal pulsating tests were carried out at a stress ratio R = 0.1. Both the 06CV and 1CSi industrial material were used to manufacture component demonstrators. The results for 06CV industrial casts bainitised at 250 °C are shown in Figure 40. A significant and unexpected decrease in fatigue strength was observed between results obtained on notched tension-tension specimens and component demonstrators.

![Pulsating tests results for 06CV industrial cast bainitized at 250°C.](image)
Investigation of broken specimens revealed that an edge at the bore intersection, as shown in Figure 41 due to manufacturing failure was identified as crack origin for all specimens shown in Figure 40.

Figure 41: SEM graph of a broken specimen at internal pulsating test for 06CV industrial cast bainitized at 250 °C. An edge due to manufacturing failure can be seen at the bore intersection causes crack initiation and a lowering of the fatigue properties.

Therefore the edge was removed and further tests carried out. As expected, the fatigue strength was significantly improved as shown by the position of the red dots in Figure 42. For the second industrial cast 1CSi the manufacturing was chosen to remove the edge from the beginning. The results for both industrial casts, 06CV and 1CSi, are shown in Figure 42.

Figure 42: Pulsating tests results for 06CV industrial cast bainitized at 250°C and 1CSi industrial cast bainitized at 250°C. Red dots (●) indicates the results for samples of 06CV-250 were the edge at the bore intersection was removed. Blue dots (●) indicates the results for 1CSi-250. Numbers of runouts on the same level are indicated in the diagram.

Comparing the obtained results with removed edge for both industrial casts, 06CV and 1CSi, the fatigue strength at 10^7 cycles of 1CSi are lower than for 06CV this corresponds with results obtained from notched fatigue specimens shown above.
7.5 CONCLUSIONS AND ACHIEVEMENTS

7.5.1 Significant deviations from work plan
There were no significant deviations from the initial work plan, except for delays carried over from the first part of the project. Some of the results were achieved in the second part of 2011.

7.5.2 Main results and conclusions
Industrial materials were tested in both wear and fatigue applications. Metso manufactured scrap shear blades using the 06CV industrial material. The durability of these blades was estimated as similar to that achieved with significantly more expensive alloys. Based on field test results, Metso estimated that a cost reduction in 10 to 20% could be achieved with the new material.

Bosch manufactured high pressure injection component demonstrators out of both industrial materials. Results obtained on 1CSi were well below expectations. This was attributed to the relatively poor cleanliness of the material. In contrast, results obtained on 06CV were on par with those obtained on bainitised 100Cr6 in spite of the significantly lower UTS of the former. Because fatigue properties were difficult to correlate to microstructure, it is expected that further improvement can be made over this first set of results, once the contributing factors are better understood and quantified.
APPENDIX A : CRITICAL COOLING RATE FOR METSO'S COMPONENT

A cube of 80x80x80 mm (largest component heat-treatable in Ascometal CREAS salt-bath installation) was prepared from a AISI 9350 billet and instrumented with 4 thermocouples as per Figure 43. This alloy was chosen for its availability and very high hardenability (to ensure transformation latent heat would not affect measurements).

![Figure 43: positions of the thermocouple in the 80x80x80 mm cube for salt-bath heat-treatments.](image)

The cube was placed in a steel basket and immersed in a salt-bath at 920 °C for 1 h. The basket was then transferred to a second salt-bath held at 250 °C (the duration of the transfer was kept as short as possible and presumably under 15 s) and kept for 3 hours.

Cooling rate calculations were carried out with Forge 2008 (Transvalor) using thermal exchange coefficients provided by Durferrit. In a first series of calculations, data for density, thermal conductivity and heat capacity for the common steel grade 42CrMo4 were used. However, as these implicitly include the effect of transformation from austenite to ferrite-pearlite (see Figure 44), they were replaced by data obtained from extrapolation of the values for austenite alone.

<table>
<thead>
<tr>
<th>Case</th>
<th>Material data</th>
<th>Heat exchange coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>$\rho$, $C_p$, $\lambda$ for 42CrMo4, unmodified</td>
<td>$h(T)$ as provided by Durferrit</td>
</tr>
<tr>
<td>II</td>
<td>$\rho$, $C_p$, $\lambda$ for 42CrMo4, modified to represent metastable austenite</td>
<td>$h(T)$ as provided by Durferrit</td>
</tr>
<tr>
<td>III</td>
<td>$\rho$, $C_p$, $\lambda$ for 42CrMo4, modified to represent metastable austenite</td>
<td>$h(T)$ modified for best fit</td>
</tr>
</tbody>
</table>

Table 33: Data used for the different calculations.

Once the material data modified, adjustments were made to the heat exchange coefficient until calculation and experiment were found to agree.

Finally, calculations were carried out for a 400x235x75 mm block (similar to METSO’s component). Table 33 summarises the conditions considered.
Figure 44: Data for steel grade 42CrMo4 (ρ, Cp, λ). The original set applies to slow cooling with ferrite-pearlite transformation. It has been modified to better represent metastable austenite.

Figure 45: Calculation results and comparisons with experimental data.

As shown in Figure 45, the comparison between calculation and measurements on the 80 mm cube shows a noticeable disagreement when using default material data and heat exchange coefficients as provided by Durferrit. When taking into account the absence of transformation (metastable austenite), the agreement is slightly improved, particularly for temperatures under 600 °C. This is expected as the modifications impact material data under the transformation temperature. After modifications of the heat exchange coefficient as a function of temperature (Figure 46), an excellent agreement is reached between experimental measurements and calculation.
As shown in Figure 46, the fitted heat exchange coefficient is significantly below that provided by Durferrit. Nevertheless, the values remain within the range of published values (see for example Techniques de l’Ingénieur, M1-126). The difference between the two sets of data may be caused by the use of a steel basket to hold the sample in the salt bath. Although the basket allows for circulation of the molten salt, it may reduce thermal exchange and therefore result in a localized heating of the molten salt. In addition, it is likely that data provided by Durferrit are for a salt bath with agitation.

Using default and fitted material and heat exchange data, the cooling rate achieved at the centre of METSO’s component (410 x 240 x 70 mm) was estimated by calculation. As shown in Figure 47, the cooling rate calculated for metastable austenite and with the fitted heat exchange coefficient is significantly lower than that calculated with default data.

It was then possible to estimate the required hardenability for the steel grade designed for METSO’s application. A critical cooling rate of 1.5 °C/s should be achieved in order to ensure absence of pearlite from the final microstructure.
APPENDIX B : ALLOY DESIGN

The theory on which the present design is based has been described elsewhere [CGM, 2003a; CGM, 2003b; FGC, 2001, FGC, 2004]. The aim of this project is to develop steels which transform to bainite at temperatures as low as the reference steels (200-350 °C) shown in Table 34, in time scales which are practical for industrial applications (less than 24 hours i.e. SP10C transformation kinetics). Low Bs and Ms temperatures can be achieved using high carbon concentration and to a lesser extent, solutes such as manganese, chromium which in the present context increase the stability of austenite relative to ferrite. The alloys should also contain sufficient silicon (at least 1.5 wt.%) to suppress the precipitation of cementite from austenite.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Co</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>AUST2</td>
<td>0.98</td>
<td>1.46</td>
<td>1.89</td>
<td>1.26</td>
<td>0.26</td>
<td>0.09</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SP9C</td>
<td>0.83</td>
<td>1.57</td>
<td>1.98</td>
<td>1.02</td>
<td>0.24</td>
<td>-</td>
<td>1.54</td>
<td>-</td>
</tr>
<tr>
<td>SP10C</td>
<td>0.78</td>
<td>1.49</td>
<td>1.95</td>
<td>0.97</td>
<td>0.24</td>
<td>-</td>
<td>1.60</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Table 34 : Compositions of reference alloys, wt%.

Figure 48 shows calculated Bs and Ms temperatures of some designed alloys together with those of the reference steels. As expected, the higher the carbon content, the lower the transformation temperatures. However, as Figure 49a illustrates, alloys with a carbon content of 1 wt.-% might exhibit slow bainite reactions since their driving force for bainite formation at low temperature is much lower than that in SP10C reference steel. For this reason, it was recommended to decrease the carbon content down to 0.8 or 0.6% C to speed up the transformation in some of the designed alloys to a level comparable to that in SP10C reference steel (see corresponding calculation in Figure 49b). Finally, the use of microalloying additions, 0.03 wt.% Nb was suggested to refine the austenite grain size and thus achieve a further rate increment during bainite formation at low temperature.

On the other hand, theory indicates that one of the largest effects on bainite plate thickness is due to the strength of the austenite. In this sense, extra additions of 0.1 wt.% Mo and 2.5 wt.% Si were recommended to increase austenite strength before transformation as neural network calculations in Figure 50 suggest.
Figure 49: Influence of carbon content on driving force for bainite formation at low temperature (200 °C).

Figure 50: Neural Network results on the effect of chemical composition on austenite strength before bainite formation. Red is for SP9C, blue for SP10C.

Table 35 shows the chemical composition of the designed steels. NANOBAIN formation will be explored for three different carbon contents (1.0, 0.8 and 0.6 wt.%) in a simple chemical composition system, Fe-C-Si-Mn-Cr. Moreover, the effect of Nb and Mo on the bainite reaction kinetics and plate thickness refinement will be investigated.
<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>1C-Nb</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>0.03</td>
</tr>
<tr>
<td>1C-Mo</td>
<td>1.00</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>0.10</td>
<td>---</td>
</tr>
<tr>
<td>1C-2.5Si</td>
<td>1.00</td>
<td>2.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.8C</td>
<td>0.80</td>
<td>1.50</td>
<td>0.75</td>
<td>0.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C-2.5Cr</td>
<td>0.60</td>
<td>1.50</td>
<td>0.75</td>
<td>2.50</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>0.6C-Nb</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>---</td>
<td>0.03</td>
</tr>
<tr>
<td>0.6C-Mo</td>
<td>0.60</td>
<td>1.50</td>
<td>1.25</td>
<td>1.50</td>
<td>0.10</td>
<td>---</td>
</tr>
</tbody>
</table>

Table 35: Chemical composition of selected alloys (wt-%).

Calculated TTT diagrams, allow estimating the hardenability of the alloy for Bosch and Metso-Minerals components. Results suggest that the formation of proeutectoid ferrite and pearlite can be avoided during the austempering treatment of Bosch and Metso-Minerals components.
One of the approaches taken during the design stage in order to increase the transformation rate was to control the previous austenite grain size (PAGS). As bainite nucleates primarily in the austenite grain boundaries, a decrease in PAGS will ensure an increase in the potent nucleation sites for bainitic ferrite [CGM, 2003b]. With this aim in mind an alloy with Nb (1CNb) was designed. Nb through its carbides exerts a control of the PAGS even at high temperatures, see theoretical calculations using MTDATA [MTDATA] in Figure 51, where the presence of Nb carbides for the 1CNb chemical composition is evident in the austenitic field.

PAGS was measured by thermal etching after austenitising at 950 °C for 15 min. This method consists in revealing the austenite grain boundaries by exposing a pre-polished specimen to a high temperature in an inert atmosphere. This leads to the formation of grooves at the intersections of the austenite grain boundaries with the polished surface. These grooves decorate the austenite grain boundaries and make them visible at room temperature in the optical microscope [CGA, 2001], see Figure 52. Results of the average measured value and distribution of PAGS are shown in Figure 53, where is evident that Nb is exerting a pining effect on the austenite grain size during austenitisation.

Table 36 summarizes the transformation times needed to finish bainitic transformation at different isothermal temperatures, showing that there is, for the higher temperatures, a decrease in the time, thus confirming the beneficial effect on the transformation kinetics of small PAGS. However in this case the
full potential of such procedure might be masked by the influence of other factors, such as the strength of austenite, i.e. chemical composition. It seems reasonable to assume that a stronger austenite will require more energy for a ferrite plate to growth i.e. slowing down the transformation. We speculate that the effect of the PAGS on the transformation kinetics might be more evident when comparing the same steel with big and small PAGS.

**Figure 53**: PAGS distribution and average value for 1C and 1CNb steels.

<table>
<thead>
<tr>
<th></th>
<th>1C</th>
<th>1C-Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>T/ºC</td>
<td>t/h</td>
<td>t/h</td>
</tr>
<tr>
<td>350</td>
<td>6.36</td>
<td>2.67</td>
</tr>
<tr>
<td>300</td>
<td>6.09</td>
<td>5.00</td>
</tr>
<tr>
<td>260</td>
<td>9.07</td>
<td>7.93</td>
</tr>
<tr>
<td>240</td>
<td>8.41</td>
<td>9.62</td>
</tr>
<tr>
<td>220</td>
<td>18.5</td>
<td>18.0</td>
</tr>
<tr>
<td>200</td>
<td>32.59</td>
<td>40.38</td>
</tr>
</tbody>
</table>

Table 36: 1C and 1CNb transformation times needed to finish bainitic transformation at different temperatures.
APPENDIX D: DESIGN OF 0.6%C INDUSTRIAL GRADE

As detailed in Appendix A, the required critical cooling rate (absence of ferrite-pearlite) was estimated to 1.5 °C/s for the industrial component to be manufactured by METSO.

For the laboratory heat however (06CNb), this critical cooling rate was about 3 °C/s. Therefore, two modified compositions were investigated with higher hardenability (Table 37).

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Al</th>
<th>Nb</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6C-Nb</td>
<td>640</td>
<td>1600</td>
<td>1270</td>
<td>16</td>
<td>13</td>
<td>160</td>
<td>1500</td>
<td>3</td>
<td>200</td>
<td>12</td>
<td>35</td>
</tr>
<tr>
<td>Target composition 1</td>
<td>650</td>
<td>1600</td>
<td>1500</td>
<td>&lt;15</td>
<td>&lt;15</td>
<td>200</td>
<td>1500</td>
<td>150</td>
<td>200</td>
<td>25</td>
<td>10</td>
</tr>
<tr>
<td>Target composition 2</td>
<td>650</td>
<td>1600</td>
<td>1250</td>
<td>&lt;15</td>
<td>&lt;15</td>
<td>200</td>
<td>1750</td>
<td>150</td>
<td>200</td>
<td>25</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 37: Composition of 06CNb and of proposed variants for industrial manufacture, in 10⁻³ wt%.

For each of these proposed compositions, the solidification path was calculated using the Scheil module integrated to the Thermocalc software, allowing for carbon back-diffusion. This allows estimation of the end of solidification temperature and of the segregation amplitude.

As shown in Figure 54, the second composition is less prone to Mn segregation than the first one. End of solidification temperatures are approximately identical (1290 °C, Figure 55), and in both compositions, precipitation of Nb(C,N) is expected in the liquid in spite of the low Nb levels (compared to the experimental 06CNb cast).

Primary niobium carbonitrides are expected to be neutral or detrimental to mechanical properties, in particular fatigue. For this reason, it was proposed not to use this element in the industrial heat, and the influence of vanadium was investigated instead. The composition proposed is therefore that shown in Table 38. As shown in Figure 56, the calculated phase stability for this composition suggests that vanadium carbides will precipitate in significant quantities below 1000 °C, and may therefore provide the grain boundary pinning effect initially sought through niobium additions (all the more since
austenitizing will typically be carried out at temperatures around 850-900 °C). The end of solidification temperature is not expected to change with this modification.

<table>
<thead>
<tr>
<th>Final target composition</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cu</th>
<th>Al</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>650</td>
<td>1600</td>
<td>1250</td>
<td>&lt;15</td>
<td>&lt;15</td>
<td>200</td>
<td>1750</td>
<td>150</td>
<td>200</td>
<td>25</td>
<td>120</td>
</tr>
</tbody>
</table>

Table 38: Proposed composition for the 06CNb based industrial heat. Nb has actually been replaced by V to avoid formation of primary carbonitrides.

Figure 56: Equilibrium phase mass fraction as a function of temperature for the proposed composition. Precipitation of V(C,N) begins for temperatures under 1070 °C but is only significant under 1000 °C.
APPENDIX E: KINETICS OF BAINITE FORMATION

Methodology

All three partners involved are similarly equipped with Adamel-Lhomargy DT1000 high resolution dilatometers and used identical procedures for CCT determination. Cylindrical specimens 3 mm in radius by 12 mm in length were used for all dilatometry measurements. In addition, CENIM and ASCOMETAL also use a Baehr 805 which is similar in principle to the DT1000 with the exception that heating is carried out using induction instead of lamps.

In the dilatometer, dimensional variations of the specimen are transmitted via an amorphous silica pushrod. These variations are measured by a LVDT sensor in a gas-tight enclosure enabling testing under vacuum or in an inert atmosphere. The temperature is measured with a 0.1 mm diameter Chromel–Alumel (Type K) thermocouple welded to the specimen. Cooling is carried out by blowing a jet of helium gas directly onto the specimen surface. The helium flow rate during cooling is controlled by a proportional servovalve. The excellent efficiency of heat transmission and the very low thermal inertia of the system ensure that the heating and cooling rates ranging from 0.003 °C/s to approximately 200 °C/s remain constant.

CCT diagrams were built by investigating the microstructure and hardness of dilatometry specimens after a heat-treatment schedule as per Figure 57.

![Figure 57: Typical heat-treatment schedule for CCT determination. Actual cooling rates may vary depending on the grade investigated.](image)

Standard metallographic sample preparation techniques for microstructural observation under optical microscopy were used. The etching agent used was 2-5% Nital.

Equipment for TTT diagram determination is identical to that used for CCT. In this case however, samples are fast cooled to the required transformation temperature before isothermal holding. This is schematically illustrated in Figure 13.

In these heat-treatments, the cooling rate CR was adapted to the steel grade investigated, to ensure absence of transformation above Tγ. It was therefore typically of 10 °C/s for the 06Cx grades, and of 30 °C/s for the 08C and 1Cx grades.

Transformation times are given, when possible, as times to reach a given percentage of the reaction (typically t0%, t50%, and t100%). The end time t100% is typically estimated for 98% transformation to avoid too large an uncertainty due to the asymptotic nature of the reaction kinetics at long times.
For practical reasons, isothermal treatments of more than \( \sim 20 \) h are difficult to carry out in the dilatometer. Therefore, in some cases, the completion of the bainite reaction could not be reached. In those cases, the \( t_{100\%} \) was estimated by extrapolation of the transformation kinetics.

It must also be underlined that \( t_{100\%} \), for example, does not imply a 100% bainitic structure, but that the bainite reaction has reached completion. Completion of the bainite reaction may lead to a microstructure with, for example, 20% residual austenite and 80% bainite.

Following the isothermal holding, samples were cooled (the cooling rate being here of no importance), and the specimens examined (metallography, hardness and in some cases, residual austenite).

**Austenitising conditions**

As explained in the main part of the report, austenitising temperatures were chosen on the basis of \( \text{Ac}_3 / \text{Acm} \) measurements, and set to 950 °C for the 1%C grades, and 900 °C for the 0.6%C grades.

**CCT diagrams**

The following present the CCT diagrams as determined for all the grades investigated in the course of the project (Figure 58 to Figure 66).

![CCT diagram for the 1C grade. A cooling rate of 20 °C/s was necessary to completely avoid the presence of grain boundary pearlite.](image)
Figure 59: CCT diagram for the 1CNb grade. A cooling rate of 20 °C/s was necessary to completely avoid the presence of grain boundary pearlite.

Figure 60: CCT diagram for the 1CMo grade. A cooling rate of 15 °C/s was necessary to avoid the formation of grain boundary pearlite.
**Figure 61:** CCT diagram for the 1CSi grade. A cooling rate of at least 10 °C/s and perhaps up to 20 °C/s was necessary to completely avoid the presence of grain boundary pearlite.

**Figure 62:** CCT diagram for 08C grade, after austenitisation at 890 °C for 15 min. A cooling rate of at least 13 °C/s was necessary to entirely suppress pearlite formation.
Figure 63: CCT diagram for 06C grade, after austenitisation at 890 °C for 15 min. A cooling rate of at least 5 °C/s was necessary to entirely suppress pearlite formation.

Figure 64: CCT diagram for the 06CCr grade. A cooling rate of at least 5 °C/s was necessary to completely avoid the presence of grain boundary pearlite.
Figure 65: CCT diagram for 06CNb grade, after austenitisation at 890 °C for 15 min. A cooling rate of 5 °C/s was necessary to entirely suppress pearlite formation (all measures by CENIM except 0.5 °C/s).

Figure 66: CCT diagram for the 06CMo grade. A cooling rate of at least 3 °C/s was necessary to completely avoid the presence of grain boundary pearlite.

Example microstructures are shown in Figure 67, illustrating a typical fully quenched microstructure (martensite and retained austenite) in the dilatometry specimen HE37 (1CNb) cooled at 20 °C/s, and the presence of traces of grain boundary pearlite in HE35 (1CMo) cooled at 17 °C/s.
Figure 67: Micrographs of dilatometry samples HE37 (1CNb), 20°C/s and HE35 (1CMo), 17°C/s. Etched in 4% Nital.

TTT Diagrams

An example of results as obtained in dilatometry is shown in Figure 68 for the 1C grade. In a number of cases for the 1C alloys, some degree of extrapolation was required to estimate the time for the end of the bainite reaction.

All results in terms of duration of the bainite reaction are shown in the main part of the report and are therefore not reproduced in this appendix (Figure 4 and Figure 5).

Examples of microstructures are shown in Figure 69 for the 1CNb alloy isothermally transformed at different temperatures.
Figure 69: Micrographs of dilatometry specimens 1CNb transformed at a) 200 °C, b) 300 °C and c) 350 °C. The microstructure is carbide free bainite with retained austenite in all cases.
APPENDIX F: ROLLING-SLIDING TESTING

Methodology

In order to replicate rolling/sliding wear conditions, two samples (discs of 45 mm diameter, 10 mm thickness) of the same composition and similar surface roughness are paired and mounted on a UTM 2000 twin disc machine for each test (Figure 70 and Figure 71). The wear tests were run in dry conditions at 100 RPM and 95 RPM respectively, creating a 5% slip between the two discs. Using such a high slip percentage is needed in order to maintain reasonable test durations, while still obtaining measurable weight losses. The applied load was set to 300 N for all tests and running time was 5 hours, all being performed at room temperature and with approximately 25% humidity. The quantitative analysis of the wear performance was then determined by measuring mass loss, wear rate and determining the specific wear rate (K). The UTM 2000 can be used for solid state friction without lubrication, or boundary and mixed conditions with liquid lubricants, which allows tests of both lubricants and materials. In dry conditions, the linear wear of both specimens as well as the friction coefficient is measured continuously. The applied load is measured with a force sensor on the load lever and the friction coefficient is calculated by measuring the traction force between the discs, using a torque sensor.

Surface Roughness of tested discs

The surface roughness of the samples was analysed by vertical optical interferometry and the results are presented in Figure 72. This was done in order to characterise the initial conditions of the contact surface. As can be seen most discs have a surface roughness (Ra) of about 0.4 µm with the exception of three groups of samples. While different surface roughness can affect the run-in period, once the initial texture of the samples has been removed by wear, the effect is minimized.
Friction behaviour

In Figure 73, the frictional behaviour of selected NANOBAIN grades and the reference material 100Cr6 austempered at 250°C is presented. As can be seen, the average friction coefficient reaches values of approximately 0.55 in all of the samples once steady state friction has been achieved. The run-in stage however, is slightly different from sample to sample. This stage depends mostly on the initial roughness of the samples.

The initial surface roughness of the tested discs ranged from 0.3 to 0.8 µm. While there is some effect of initial roughness on wear, it has been determined in a previous study [ALE, 2011] that, after five hours of testing, the effect of initial roughness on wear is negligible.

A friction coefficient of 0.1 is common at the start of the tests. Subsequently, the friction increases as the two surfaces are “run in”. It is known that the steady state friction coefficient for steel sliding against steel in dry conditions is in the range of 0.5-0.7 which is similar to the observed steady state
friction coefficients in the NANOBAIN grades. This is because the adhesive forces acting in the contact are highly dependent on the atoms present at asperity-asperity contact points and less dependent on the microstructure which generally has a higher scale. Since the NANOBAIN grades are similar to common steels (with regard to their chemical composition), the friction level are expected to be similar to that of other conventional steels.

Wear rate of Nanobain grades
The specific wear rate was estimated from the mass loss as measured on each disc, and converted into volume loss.

\[ SWR = \frac{\text{Volume loss (disc1+disc2)}}{\text{Sliding distance} \times \text{Load}} \]

The results are presented and discussed in the core of the report (Figure 24).

Analyses of worn surfaces
Figure 74 through Figure 82, show SEM images taken from the samples surface after rolling-sliding tests for 5 hours. In these figures, the sliding force has been directed upwards. The images are taken at the same magnification in order to allow direct comparison. Through analysis the surface characteristics it has been possible to determine three main damage types on the samples:

Accumulation of oxidized wear debris: due to the high sliding ratio at which the tests are done (5%), it is likely that the high adhesive forces present are responsible for the production of the wear debris at shorter test times. Once produced, the debris is oxidized and due to the geometry of the contact it can be pressed into the contact repeatedly. This causes the accumulation of debris which can vary from sample to sample. In some cases the surface presents what appear to be abrasion marks but this happens at a small scale and they are not found in all samples.

Delamination: This mechanism has been found in all of the studied samples and is thought to be the main mechanism of material loss in all cases, including the reference steel (100Cr6). This is the likely result of rolling contact fatigue cracking. It is known from literature that cracks that grow under RCF, will either nucleate at the surface or below the surface depending on the friction levels at the contact. Once a crack is nucleated in the surface, it will tend to grow into the bulk of the material initially, afterwards parallel to the surface and finally back towards the surface until the whole flake is removed.
Indentation marks: this type of damage is not clearly present in all the samples. In Figure 74 and Figure 75, the debris accumulation is higher than in the other samples and possibly has covered any indentation marks present on the surface. However in the rest of the samples (Figure 76 to Figure 82), indentation marks are clearly present. These indentations could be the result of two different wear mechanisms. It is possible that wear debris accumulated in the surface agglomerated in such a way that when it came into contact it caused plastic deformation of the counter surface resulting in the observed features. Another possibility is that the marks are the result of flakes of material that were removed from the surface.
Figure 78: SEM images at the surface of sample 08C250 after wear testing.

Figure 79: SEM images at the surface of sample 08C270 after wear testing.

Figure 80: SEM images at the surface of sample 1C-950AT after wear testing.

Figure 81: SEM images at the surface of sample 1CSi after wear testing.
As can be observed in Figure 74 to Figure 82, the surface features of all worn samples have very similar features. This suggests that the mechanisms operating to remove material during the tests are the same in all cases.

**Examination of cross-sections**

Figure 83 and Figure 84 provide an example of cross-section as were carried out and examined for all RS specimens. The micrographs present the typical damage modes observed in all the disc test specimens. These include deformation of the microstructure in the direction of sliding and delamination or “flaking”. The deformed microstructure is oriented in the direction of sliding which has also been previously observed for pearlitic rail steels. In the case of pearlitic steels, the alignment and refinement of the cementite plates, along with the deformation of the surface are considered to be a reason for their successful performance in rolling-sliding wear [AJPU, 1993]. In carbide-free steels there is no cementite, however, but there is high-carbon retained austenite, which can transform to martensite under strain. This transformation, in addition to the deformation hardening, causes the hardness to increase considerably at the contact surface [ALe, 2011].
From the cross sectional images observed, the mechanism proposed in the previous contribution has been improved. In Figure 83, it is possible to observe two delaminating flakes in which cracking has clearly started from a preferential site in the surface. In Figure 84, it is possible to observe the formation of a crack initiation site in the early stages. The microstructure around it clearly follows the contour of the defect, suggesting that deformation of an asperity has been the cause for this particular feature.

Sub-surface cracking along with surface cracking is observed in several samples. This suggests that cracking is also generated at defects located in the subsurface region of the samples where the stresses are sufficiently high to cause rolling contact fatigue cracking.

**Hardness increase near the surface layer, retained austenite**

All specimens exhibited a sharp hardness increase in the vicinity of the contact surface, as illustrated in Figure 85. The large uncertainty in these measurements (50g load) means that no significant and systematic differences could be identified between the different grades.

![Figure 85](image_url)

**Cleanliness of laboratory heats vs industrial heats**

To confirm whether differences in wear resistance between laboratory and industrial heats of Nanobain grade might be partly or fully explained by differences in cleanliness, inclusions were characterised using an instrumented optical microscope. This allows characterisation of surface in the range 30-50 mm². Minimum size for recording was set to 10 µm, all inclusions detection were identified manually.
As shown in Table 39, there are significant differences in oxygen and sulphur content between laboratory and industrial casts.

<table>
<thead>
<tr>
<th></th>
<th>S</th>
<th>O</th>
<th>Ti</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>06C (lab. cast)</td>
<td>160</td>
<td>21</td>
<td>30</td>
<td>70</td>
</tr>
<tr>
<td>06CV (industrial)</td>
<td>20</td>
<td>4</td>
<td>30</td>
<td>75</td>
</tr>
</tbody>
</table>

Table 39: Chemical composition for S, O, Ti and N as measured by optical emission spectrometry and LECO analysis. All results in ppm.

Results are shown in Figure 86. As expected from the differences in chemical compositions, the density of both oxides and sulphides were much higher in the laboratory casts. In particular, oxides alignments up to 1mm in length were found in the 06C rolling sliding specimen (Figure 87).

Figure 86: Distribution of inclusions in both investigated specimens, a) oxides and b) sulphides.

Figure 87: Large oxides alignment in 06C (lab cast) rolling-sliding specimen.
APPENDIX G : HIGH PRESSURE ABRASION AND IMPACT TESTS

The specimens for abrasion and impact test were delivered to Metso in heat treated condition. No final machining was needed after heat treatment for the wear test samples because they were subjected to run-in wear at the beginning of the test. Impact test samples were machined further to V-notch Charpy impact test samples because the un-notched samples were too tough for impact tests to get reliable results.

Charpy impact toughness tests were performed by Charpy impact hammer with 300 N load. Samples were V-notched according to impact testing standard. Tests were completed with three sample series of each composition and heat treatment.

The background for this experimental setup was to design a wear environment simulation, which combines a compressive crushing process and sideways movement. This is created by a specimen, which is fixed on a pneumatic cylinder and pressed on loose gravel bed in a rotating container (Figure 88). As the specimen is pressed down with a certain force, the abrasive particles are crushed between the sample and the rotating counterbody. After 2,5 seconds compression the specimen is lifted up automatically and a new cycle begins after another 2,5 seconds. There is a small cap between the two metal bodies to avoid friction from metal-metal contact. In this study the tests were done with a compression force of 200N. The total testing time for a sample was a wear contact of 30 minutes. Performance is measured in terms of mass loss of the specimens.

Figure 88 : High-stress abrasion test device.
APPENDIX H : PREPARATION FOR 06CV INDUSTRIAL CAST

Because of the significant difference between 06CNb (selected model alloy) and the proposed composition for the industrial heat, it was decided to manufacture one additional laboratory heat to confirm the critical cooling rate and isothermal transformation kinetics on the new composition.

The critical cooling rate was estimated by continuous cooling of dilatometric samples at different rates in a Bähr dilatometer, followed by metallographic examination of the thus heat-treated specimens.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Nb</th>
<th>Critical CR</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6C-Nb</td>
<td>0.640</td>
<td>1.600</td>
<td>1.270</td>
<td>0.016</td>
<td>0.013</td>
<td>0.160</td>
<td>1.500</td>
<td>0.003</td>
<td>-</td>
<td>0.035</td>
<td>3-5°C/s</td>
</tr>
<tr>
<td>Metso Lab cast</td>
<td>0.689</td>
<td>1.451</td>
<td>1.183</td>
<td>0.026</td>
<td>0.012</td>
<td>0.202</td>
<td>1.757</td>
<td>0.145</td>
<td>0.119</td>
<td>0.000</td>
<td>1.5°C/s</td>
</tr>
<tr>
<td>Indus min</td>
<td>0.625</td>
<td>1.580</td>
<td>1.230</td>
<td>0.000</td>
<td>0.000</td>
<td>0.180</td>
<td>1.700</td>
<td>0.145</td>
<td>0.110</td>
<td>0.000</td>
<td>-</td>
</tr>
<tr>
<td>Indus max</td>
<td>0.675</td>
<td>1.700</td>
<td>1.350</td>
<td>0.010</td>
<td>0.010</td>
<td>0.250</td>
<td>1.800</td>
<td>0.165</td>
<td>0.150</td>
<td>0.000</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 40: Composition of laboratory cast manufactured to investigate the newly proposed alloy (Metso Lab cast), in comparison with 06CNb. All values in wt%.

As shown in Table 40, the laboratory heat manufactured for that purpose had a critical cooling rate of 1.5 °C/s (as required given the dimensions of Metso’s component). Isothermal heat-treatment were carried out in a Bähr dilatometer, and indicated that end of transformation time at 250 °C was similar if not faster for the proposed composition compared to 06CNb (Table 41).

<table>
<thead>
<tr>
<th>Cast</th>
<th>Austénitization</th>
<th>HT</th>
<th>Time to transform</th>
<th>Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6CNb</td>
<td>890°C/30 min</td>
<td>250°C</td>
<td>10 h</td>
<td>604 HV</td>
</tr>
<tr>
<td>Metso Lab cast</td>
<td>890°C/30 min</td>
<td>250°C</td>
<td>9.3 h</td>
<td>620 HV</td>
</tr>
</tbody>
</table>

Table 41: Time for isothermal bainite transformation and final hardness for 06CNb and the proposed composition.

Finally, it was important to verify hot workability of the material in order to avoid manufacturing difficulties, particularly during reheating before hot-rolling.

For that purpose, hot-workability tests were carried out on specimens machined from the 06CNb lab casts. The vanadium lab casts could not be used because the available quantities were insufficient. It is thought, however, that the modifications made will not affect significantly hot-workability (carbon and silicon amounts being kept identical).
Figure 89: Striction vs test temperature for the 06CNb grade. These results highlight the need to avoid heating beyond 1200 °C, and pay close attention to the hot-rolling end temperature.

In these tests, reduction of area is measured for tensile tests carried out at different temperatures. The temperature interval in which reduction of area exceeds 50% is generally considered safe for hot-rolling. In contrast with a conventional high temperature tensile test, the test temperature is reached after an initial isothermal holding at 1200 °C. This first step leads to a coarse microstructure, as would be obtained on a solidification microstructure, or after reheating of ingots. As shown in Figure 89, the results underline the need to avoid heating above 1200 °C, while ensuring that temperature at the end of hot-rolling does not drop below approx. 900 °C.
APPENDIX I : SIDENOR TENSILE TESTING ON 1CSI-INDUS

Mechanical properties on austempered samples (220°C, 250°C, 280°C and 310°C) were characterised. The samples were austenitized at 950°C for 60 minutes in inert atmosphere and then quenched in salt bath (austempered) in the following way:

- 22 hours @ 220°C
- 16 hours @ 250°C
- 16 hours @ 280°C
- 16 hours @ 310°C

Finally, they were finished by hard machining and V-notch by electro-discharge machining. The results are shown in Table 42 and Figure 90.

<table>
<thead>
<tr>
<th>TTT (°C)</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>Elong (%)</th>
<th>RofA (%)</th>
<th>Kv (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22h @ 220</td>
<td>2125,5</td>
<td>1610,7</td>
<td>6,4</td>
<td>7,8</td>
<td>2,8</td>
</tr>
<tr>
<td>16h @ 250</td>
<td>1860,6</td>
<td>1556,8</td>
<td>7,2</td>
<td>17,8</td>
<td>5,0</td>
</tr>
<tr>
<td>16h @ 280</td>
<td>1831,2</td>
<td>1525,6</td>
<td>9,1</td>
<td>23,7</td>
<td>7,5</td>
</tr>
<tr>
<td>16h @ 310</td>
<td>1627,9</td>
<td>1236,9</td>
<td>14,4</td>
<td>18,4</td>
<td>8,7</td>
</tr>
</tbody>
</table>

Table 42 : Mechanical properties in austempered condition at different temperatures.

Figure 90 : Tensile tests curves of 100SiCr10-4 steel.

Microstructure in austempered condition was nanobainite, showing some segregation bands with high content of blocky retained austenite. Bainitic ferrite plate thickness was measured to compare to experimental grade results and to evaluate isothermal temperature dependence. With values ranging from 36 to 45 nm, there did not appear to be a strong correlation between isothermal transformation temperature and lath thickness.
APPENDIX J : FATIGUE TESTING ON INDUSTRIAL CASTS

Notched tension tension specimens
Notched conditions selected here are representative of the functioning of heavily loaded diesel injection components. The geometry of the notched specimen is shown in Figure 91.

Figure 91 : Specimen geometry for notched fatigue testing on industrial heats.

The manufacturing process of both specimens is soft machining, salt bath heat treatment and hard machining.

The fatigue tests for notched conditions were carried out with the stress ratio \( R = 0.1 \) to test the stress ratio close to application such like diesel injection system. Tests were carried out in pulsating machine at a frequency of 150 Hz and with a limit set to \( 10^7 \) cycles. These statistical values were obtained by distinguishing between different failure mechanisms, which were identified by SEM examination of the specimen fracture surface after testing.

Results on 06CV bainitised at 270 °C are shown in Figure 92. The failure modes are indicated within the diagram. Two failure modes are present: crack initiation from the surface (see Figure 93) and crack initiation from non metallic inclusion beneath the surface (see Figure 94). A division in this two different failure modes can be clearly identified. Early cracking (< \( 10^5 \) cycles) at higher stresses typically initiate from the surface, and later cracking (> \( 10^6 \) cycles) at slightly lower stress levels, on inclusions. This division is typical for high strength steels at that level of tensile strength.

Figure 92 : The fatigue results of the industrial cast 06CV bainitised at 270°C (06CV-270-FAT).
Fatigue crack originate from surface.

Figure 93: Typical SEM pictures for crack starting from the surface for 06CV-270-FAT.

Fatigue crack originate subsurface from inclusion (TiCN).

Figure 94: Typical SEM pictures for crack starting from a Ti(C,N) beneath the surface for 06CV-270-FAT.
Figure 95: The fatigue results of the industrial cast 06CV bainitised at 250°C (06CV-250-FAT); tested in notched condition, $K_t = 2.0$ and with a stress ratio of $R = 0.1$.

Figure 95 shows the fatigue results for the industrial cast 06CV bainitised at 250 °C. As for 06CV-270-FAT, a division due to the change in failure mode is clearly identified. In comparison with material heat-treated at 250 °C, the 06CV material exhibited a slight increase in its $10^7$ fatigue limit for a heat-treatment at 270 °C; this increase however was not found to be statistically significant.

Figure 96: The fatigue results of the industrial cast 1CSi bainitised at 220°C (1CSi-220-FAT), tested in notched condition, $K_t = 2.0$ and with a stress ratio of $R = 0.1$.

Fatigue results for 1CSi industrial cast are not as readily interpreted as those for 06CV. In Figure 96 the fatigue results of the industrial cast 1CSi bainitised at 220 °C (1CSi-220-FAT) are shown. Due to a high scatter in test data, all samples were investigated using SEM. In addition to initiation sites as already reported, two cracks were found to have originated from corrosion pits. Large cleavage fracture areas within the fatigue crack areas could be found as shown in Figure 97.
Figure 97: SEM graph for 1CSi-220-FAT specimen tested as a stress level of $S_a = 280$ MPa and broken after $N = 642,000$ cycles.

Figure 98 shows the fatigue results of the industrial cast 1CSi bainitised at 250°C (1CSi-250-FAT). The failure mode is indicated within the diagram. Compared to 1CSi-220-FAT the scatter is reduced due to an increase of the ductility of the material by more than twice (see Task 6.1). A division within two different failure modes can be seen, but this division is not as clear as in the case of 06CV.

![Graph showing fatigue results](image)

**Figure 98**: The fatigue results of the industrial cast 1CSi bainitised at 250°C (1CSi-250-FAT); tested in notched condition, $K_t = 2.0$ and with a stress ratio of $R = 0.1$.

Compared with 1CSi-220-FAT, the same fraction of cleavage fracture areas were observed, as shown in Figure 99.

![SEM micrograph](image)

**Figure 99**: SEM micrograph of (a) 1CSi-250-FAT specimen tested at $S_a = 280$ MPa and broken after 73,000 cycles and (b) 1CSi-220-FAT specimen tested at $S_a = 280$ MPa and broken after 642,000 cycles (b).
APPENDIX K: METALLOGRAPHIC EXAMINATION OF 06CV INDUSTRIAL COMPONENT

Following salt-bath heat-treatment, one of the components manufactured by METSO was cut following Figure 100, and specimens were sent to ASCOMETAL for metallographic examination.

Metallographic examinations (hardness, retained austenite and bainite lath thickness) were measured at all points indicated in Figure 101, for a section taken near the component surface, and for one taken at its centre (Figure 100). As shown in Table 43, hardness was remarkably homogeneous throughout the component, thus demonstrating that transformation took place uniformly at the bainitising temperature, and not during cooling. Bainite lath thickness was measured at the centre and found to be 58 nm.

<table>
<thead>
<tr>
<th>Surface</th>
<th>Side</th>
<th>Middle</th>
<th>Core</th>
<th>Surface</th>
<th>Side</th>
<th>Middle</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>545</td>
<td>535</td>
<td>542</td>
<td>541</td>
<td>541</td>
<td>543</td>
</tr>
</tbody>
</table>

Table 43: 30 kg load Vickers hardness for METSO's component. The hardness did not vary significantly with position.
APPENDIX L: FIELD TESTING OF COMPONENT MANUFACTURED BY METSO

Test machine: Scrap shear EC 1034-10
Blade dimensions: 524 x 80 x 200mm
Test (outside) temperature 0 – 15 °C
Number of cuts (one side): 4965
Average number of cuts with normal blades (one side): 8000-12000

Figure 102: EC 1034-10 machine in Karjaa, Finland

Test started with (<6mm carbon steel) and after 58 cuts they changed to >6mm carbon steel. After 2500 cuts the material was changed to old railway rails (100cuts). Last 2300 cuts was stainless steel. Below are presented some pictures and comments in different stages of the test.

0 cuts: The upper blades set is installed (Figure 103).

Figure 103: Installed upper blade set (0 cuts)

58 cuts: Upper and lower blade set exhibited the same kind of small rounding as can be seen with standard blade set. No major dents can be seen. Material cut was <6mm carbon steel.

Figure 104: Upper blade rounding can be seen on the left side. Lower blade also shows no major problems with the edge rounding (right).
122 cuts: Change from <6mm to >6mm carbon steel did not show any major effect in the wear rate of the blade. Some small dents on the edges in upper and lower blade can be seen but no big changes in blades.

![Figure 105: Blade after 122 cuts looks to be in good condition (figure 105 upper and figure 106 lower). Same kind of edge rounding can be seen as in previous pictures.](image)

308 cuts: Blades seem to be in good condition. More small cracks are appearing in the cutting edge but no major dents.

![Figure 106: After 308 cuts the blade is in good condition. No major problems with the blades.](image)
502 cuts: Upper blade seems to be in good condition. Lower blade has the first bigger crack (Figure 107). It seems that there has been a brittle fracture in the edge. 10mm of material is removed from the upper surface and 20 mm from the lower surface. The crack surface has a flake like surface structure. Temperature of the lower blade was 20 °C and upper blade it was 29 °C.

![Image of 502 cuts](image)

Figure 107: After 502 cuts the upper blade looks to be fine. Lower blade (right side) is showing one bigger crack.

2500 cuts: After 2500 cuts there was a change in scrap material. 15 000t of railway rails were cut (~100 cuts). No pictures available at this phase. Usually railway rails are cut at the end before the blades are changed but for some reason this time the rails were cut already after couple of working days. That caused some major cracks on the test blades.

![Image of 2500 cuts](image)

Figure 108: After 2600 cuts (railway rails) the blades had more significant damage on the edges. There were several bigger cracks on the cutting edges.

2600 cuts: After 2600 cuts the blades were damaged in several places. Blade edges had brittle cracks. Biggest crack was seen in the first corner of upper blade (Figure 108). Before the railway rails the upper

![Image of 2600 cuts](image)
blade was not significantly damaged but after the 100 cuts the biggest damages could be observed from the corner of the upper blade. The upper blade was damaged more significantly by the railway rails. After the railway rails scrap material was changed to stainless steel.

3762 cuts: After 3762 cuts bigger cracks were seen on upper blade surface. Also on the lower blade there were some bigger cracks and edge rounding. Scrap material used was stainless steel. It was obvious that the material was more difficult to cut compared to carbon steel. Temperature of the upper blade was 64 °C after testing. Stainless steel scrap was damaging the blade that was already damaged from the railway rails.

4965 cuts: The test was finished after 4965 cuts. The blade was not cutting well enough anymore. 100 cuts with railway bars and 2300 cuts with stainless steel caused the blades to be worn out. Blades are usually changed always after a bigger amount of stainless steel and now the situation was the same. Blades were changed after a big amount of stainless steel scrap.
Conclusions: The blades were tested in difficult environments. First with normal scrap the blades were working well. Then after railway bars some cracks were seen. Usually railway bars are always cut just before changing the blades since they always cause cracks but this time the bars were cut in the middle of the test. Also after cutting a bigger amount of stainless steel the blades are changed. This happened also with the test blades. The test blades lasted about 0.5 times compared to normal blades but the material used in the test was difficult and also cutting was done in different order than usual. Nanobain blades can last longer time if used in optimal way. More tests have to be conducted to obtain reliable results.
APPENDIX M : NOTCHED ROTATION BENDING FATIGUE TESTING

Specimen geometry

It is well established that high cycle fatigue performances of high strength material (UTS > ~1200 MPa) is strongly influenced by defects present in the material bulk or surface. Because cleanliness of laboratory casts is not controlled, it was decided to use notched rotation bending specimens for investigations to be carried out on samples taken on such material. The combination of rotation bending method and notching would significantly limit the sampling volume since, first, the rotation bending specimens are only at the maximum stress in their outer thickness and second, the notch will further localise the volume under load along the sample length.

The notch geometry was the same for all partners, although testing facilities differences mean that length will vary slightly. The stress concentration factor was calculated by FEM methods to $K_t = 1.6$ (Figure 113).

![Figure 113 : Stress concentration factor calculated by FEM methods to $K_t = 1.6$.](image)

Manufacturing of test specimens

Soft machining, heat treatment, hard machining and testing was distributed among the partners as shown in Table 44.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Soft Machining</th>
<th>Heat Treatment</th>
<th>Hard Machining</th>
<th>Testing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CNb-220-FAT</td>
<td>ASCO</td>
<td>ASCO</td>
<td>BOSCH</td>
<td>ASCO</td>
</tr>
<tr>
<td>06C-250-FAT</td>
<td>BOSCH</td>
<td>ALD</td>
<td>BOSCH</td>
<td>CENIM</td>
</tr>
<tr>
<td>08C-270-FAT</td>
<td>BOSCH</td>
<td>BOSCH</td>
<td>BOSCH</td>
<td>ASCO</td>
</tr>
<tr>
<td>1CSi-ind-220-FAT</td>
<td>LTU</td>
<td>BOSCH</td>
<td>BOSCH</td>
<td>LTU</td>
</tr>
<tr>
<td>1CSi-ind-250-FAT</td>
<td>BOSCH</td>
<td>BOSCH</td>
<td>BOSCH</td>
<td>LTU</td>
</tr>
<tr>
<td>06CV-ind-270-FAT</td>
<td>CENIM</td>
<td>ASCO</td>
<td>BOSCH</td>
<td>ASCO</td>
</tr>
</tbody>
</table>

Table 44 : Details of the fatigue tests organisation

To obtain a comparable surface state all hard machining was carried out by the same partner (BOSCH).

Results

Results obtained on rotation bending specimens are shown in Figure 114 to Figure 122. The fatigue strength thus determined are summarized in Table 45. Because different laboratories were involved in
the actual testing, a round robin was carried out and discussions took place to ensure results may be compared.

<table>
<thead>
<tr>
<th>Condition</th>
<th>UTS / MPa</th>
<th>Sa, 50% at $10^7$ cycles / MPa for R = -1, $K_t = 1.6$ by rotation bending</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CNb-220-FAT</td>
<td>2073</td>
<td>430</td>
</tr>
<tr>
<td>06C-250-FAT</td>
<td>2023</td>
<td>665</td>
</tr>
<tr>
<td>08C-270-FAT</td>
<td>2036</td>
<td>440</td>
</tr>
<tr>
<td>1CSi-ind-220-FAT</td>
<td>2224</td>
<td>550</td>
</tr>
<tr>
<td>1CSi-ind-250-FAT</td>
<td>2072</td>
<td>535</td>
</tr>
<tr>
<td>06CV-ind-250-FAT</td>
<td>2003</td>
<td>690</td>
</tr>
<tr>
<td>06CV-ind-270-FAT</td>
<td>1822</td>
<td>675</td>
</tr>
</tbody>
</table>

**Table 45**: $10^7$ cycles fatigue strength for all conditions investigated.

![Graph](image)

**Figure 114**: The fatigue results of the lab cast 1CNb bainitised at 220°C (1CNb-220-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.
Figure 115: The fatigue results of the lab cast 06C bainitised at 250°C (06C-250-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.

Figure 116: The fatigue results of the lab cast 08C bainitised at 270°C (08C-270-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.
Figure 117: The fatigue results of the industrial cast 1C1Si bainitised at 220°C (1C1Si-ind-220-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.

Figure 118: Detail of the fatigue results of the industrial cast 1C1Si bainitised at 220°C (1C1Si-ind-220-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending. Numbers of runous on the same level are indicates by numbers.
Figure 119: The fatigue results of the industrial cast 1CSi bainitised at 250°C (1CSi-ind-250-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.

Figure 120: Detail of the fatigue results of the industrial cast 1CSi bainitised at 250°C (1CSi-ind-250-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending. Numbers of runous on the same level are indicates by numbers.
Figure 121: The fatigue results of the industrial cast 06CV bainitised at 250°C (06CV-ind-250-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.

Figure 122: The fatigue results of the industrial cast 06CV bainitised at 270°C (06CV-ind-270-FAT); tested in notched condition, $K_t = 1.6$ and with a stress ratio of $R = -1$ by rotation bending.

Analysis: example of 1CSi

It was possible to identify that all of the 1CSi220 samples presented inclusions as a starting point of fatigue cracking. The composition of the inclusions varied as shown in Figure 123 to Figure 126.
<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>6.62</td>
<td>21.87</td>
</tr>
<tr>
<td>Si K</td>
<td>0.77</td>
<td>1.09</td>
</tr>
<tr>
<td>S K</td>
<td>11.82</td>
<td>14.62</td>
</tr>
<tr>
<td>Ti K</td>
<td>38.62</td>
<td>31.97</td>
</tr>
<tr>
<td>V K</td>
<td>2.09</td>
<td>1.62</td>
</tr>
<tr>
<td>Cr K</td>
<td>3.22</td>
<td>2.46</td>
</tr>
<tr>
<td>Mn K</td>
<td>18.76</td>
<td>13.54</td>
</tr>
<tr>
<td>Fe K</td>
<td>18.08</td>
<td>12.84</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

Figure 123 : Inclusion found in the starting point of Sample 4 (593MPa, 3E6 Cycles) Grade 1CSi220

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O K</td>
<td>37.16</td>
<td>54.39</td>
</tr>
<tr>
<td>Mg K</td>
<td>4.90</td>
<td>4.72</td>
</tr>
<tr>
<td>Al K</td>
<td>16.03</td>
<td>13.91</td>
</tr>
<tr>
<td>Si K</td>
<td>8.27</td>
<td>6.89</td>
</tr>
<tr>
<td>S K</td>
<td>4.47</td>
<td>3.26</td>
</tr>
<tr>
<td>Ca K</td>
<td>27.74</td>
<td>16.21</td>
</tr>
<tr>
<td>Fe K</td>
<td>1.43</td>
<td>0.60</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

Figure 124 : Inclusion found in the starting point of Sample 6 (581MPa, 5,9E6 Cycles) Grade 1CSi220

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>1.49</td>
<td>4.17</td>
</tr>
<tr>
<td>O K</td>
<td>4.11</td>
<td>8.62</td>
</tr>
<tr>
<td>Al K</td>
<td>1.08</td>
<td>1.34</td>
</tr>
<tr>
<td>Si K</td>
<td>0.28</td>
<td>0.34</td>
</tr>
<tr>
<td>S K</td>
<td>39.71</td>
<td>41.50</td>
</tr>
<tr>
<td>Ca K</td>
<td>50.98</td>
<td>42.63</td>
</tr>
<tr>
<td>Fe K</td>
<td>2.34</td>
<td>1.41</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

Figure 125 : Inclusion found in the starting point of Sample 8 (568 MPa, 4,1E5 Cycles) Grade 1CSi220

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>6.32</td>
<td>19.78</td>
</tr>
<tr>
<td>Si K</td>
<td>1.47</td>
<td>1.09</td>
</tr>
<tr>
<td>S K</td>
<td>11.82</td>
<td>14.62</td>
</tr>
<tr>
<td>Ti K</td>
<td>38.62</td>
<td>31.97</td>
</tr>
<tr>
<td>V K</td>
<td>1.72</td>
<td>1.62</td>
</tr>
<tr>
<td>Cr K</td>
<td>3.22</td>
<td>2.46</td>
</tr>
<tr>
<td>Mn K</td>
<td>18.76</td>
<td>13.54</td>
</tr>
<tr>
<td>Fe K</td>
<td>18.08</td>
<td>12.84</td>
</tr>
<tr>
<td>Totals</td>
<td>100.00</td>
<td></td>
</tr>
</tbody>
</table>

Figure 126 : Inclusion found in the starting point of Sample 10 (556 MPa, 4,7E6 Cycles) Grade 1CSi220
Thus, part of the scatter in results can be explained by the initiations on inclusions. Another important factor affecting scatter may be the amount of intergranular cracking observed in the samples. Figure 127 and Figure 128 present fractographs from different 1CSI220 samples. As shown, they contain a large amount of intergranular fracture. Moreover, the shape of the grains suggests that they are retained austenite grains, possibly blocky retained austenite.

Figure 127: Crack initiation zone of sample 3 (593 MPa, 2.9E6 Cycles) Grade 1CSI220. The arrows point to particular features later identified as intergranular crack growth.

Figure 128: Fracture surface of sample 4 (593 MPa, 3E6 Cycles) Grade 1CSI220.
APPENDIX N : STANDING CONTACT FATIGUE TESTING

Standing contact fatigue testing
The mechanism of standing contact fatigue has been developed and studied at the Royal Institute of Technology in Stockholm from 1997 to around 2003 and is mostly based on the fretting effect. The mechanism of fretting is affected by the load, amplitude, frequency, number of cycles, temperature and humidity.

The difference in the position of the cracks (inside or outside the indent) is caused by different contact conditions between the surfaces of the indenter and the sample. The friction depends on the oscillation of the indenter and the adhesive forces between the indenter-surface and the sample-surface. Figure 129 shows a schematic of the SCF contact used for these tests.

Figure 129 : Contact between a spherical indenter and a flat object.

The strength and ductile behaviours of the two materials affect the oscillation of the indenter. In case of full slip, the cracks can occur anywhere inside the contact area. However, if the slip is only partial like shown in Figure 129, the cracks tend to occur at the edge between the slip and the non-slip-region. Slip is caused due to slightly different elastic behaviour of the materials during the cycles in case a different material is used for sample and indenter. Using the same material on the indenter and the sample minimizes the slip.

Samples preparation

All the heat treatments have been carried out by ALD, except for the 1%C sample. The initial heat treatment of all the samples was done at an austenitisation temperature of 850°C. This temperature was believed to be too low to allow the complete dissolution of carbides in the 1%C sample. Therefore, the 1% C nanobainitic sample has been heat treated a second time at LTU in a salt bath. The heat treatment consisted of austenitising the samples at 950°C for 15-20 minutes min and then quenching in a salt bath at the austempering temperature of 250°C for 8 hours.

The standing contact fatigue tests have been performed on 5 nanobainitic steels and 2 reference steels. Table 46 shows an overview of the tested materials including their heat treatment.
The standing contact fatigue (SCF) tests were carried out on circular discs of 6 to 10 mm thickness and 45 mm diameter under room temperature and without any lubrication. The tested surfaces have been ground, polished with diamond paste down to 1 micron and finally finished with a colloidal silica suspension. For these tests the crack identification has been done by optical microscopy and having the surfaces polished makes it much easier to detect the fatigue cracks. Figure 130 shows an overview of the SCF sample before and after several tests had been done on the surface.

**Figure 130 : Samples before and after (with circular indent prints) standing contact fatigue (SCF) tests.**

### Table 46 : Overview of all tested materials on standing contact fatigue. Bainitic steel is 40CrSi8 in main report, and pearlitic is 60CrSi9.

<table>
<thead>
<tr>
<th>Name</th>
<th>Steel</th>
<th>Austenisation</th>
<th>Bainitising</th>
<th>Done by</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C-250</td>
<td>1C</td>
<td>1h@950°C</td>
<td>8h@250°C</td>
<td>LTU</td>
</tr>
<tr>
<td>06C-250</td>
<td>06C</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>06CNb-250</td>
<td>06CNb</td>
<td>1h@890°C</td>
<td>12h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>08C-220</td>
<td>08C</td>
<td>1h@890°C</td>
<td>22h@220°C</td>
<td>ALD</td>
</tr>
<tr>
<td>08C-250</td>
<td>08C</td>
<td>1h@890°C</td>
<td>16h@250°C</td>
<td>ALD</td>
</tr>
<tr>
<td>Bainitic Steel (0,4% C)</td>
<td>provided by METSO</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pearlitic Steel (0,6% C)</td>
<td>provided by METSO</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The standing contact fatigue (SCF) tests were carried out on circular discs of 6 to 10 mm thickness and 45 mm diameter under room temperature and without any lubrication. The tested surfaces have been ground, polished with diamond paste down to 1 micron and finally finished with a colloidal silica suspension. For these tests the crack identification has been done by optical microscopy and having the surfaces polished makes it much easier to detect the fatigue cracks. Figure 130 shows an overview of the SCF sample before and after several tests had been done on the surface.

**Standing Contact Fatigue testing**

Figure 131 shows images from the SCF tester used. The standing contact fatigue test consists on the low cylinder applying cyclic stresses that stress the upper surface of the sample against a static spherical indenter. The indenter is a hardened steel ball that has a hardness of about 960 HV. After the tests, there is an indent left in the surface as can be seen in Figure 130. The machine can apply loads up to 25 KN under frequencies up to 60 Hz which allows high cycle testing in a relatively short time.

**Figure 131 : The SCF machine**
All the tests were carried out at a frequency of 50 Hz with 3 different amplitudes: 15 kN, 11 kN and 9 kN. The machine starts by preloading the sample to 0.5 kN which is considered the minimum load. Then the machine starts, oscillating at the preset amplitude. The criteria used in these tests consisted on the visual detection of cracks. It was tried in previous works to use microphones to detect cracking, but when the machine is operating at 50 Hz the vibrations are so high that the microphones cannot detect the appearance of the cracks. To evaluate the tests and in order to check when the cracks appear in each sample, the indents have been observed using the optical microscope at two different magnifications (x100) (x50). After each test the sample is controlled optically and based on the result (crack or no crack) a new test was started with either lower (in case of presence of cracks) or higher cycles (in case of absence of cracks) in order to find out the fatigue life at a given load (number of cycles at which the cracks appear). Once the number of cycles that produces cracking has been found the test is repeated once more to ensure the result. In each case, the top view of the indent shows that we obtain only radial cracks that go out perpendicularly from the circumference of the indent.

Results: Fatigue Crack observation

In all the nanobainitic samples studied, only radial cracks have been found and therefore, the formation of these cracks has been the criterion used in the estimation of the fatigue life. For the bainitic and pearlitic reference samples provided by Metso, no radial cracking was found, even after 1,8 million cycles at 15 kN. Table 47 presents the compiled results for the fatigue life of the nanobainitic samples tested so far.

It has been possible to plot the number of cycles to failure, i.e. the minimum number of cycles at which cracks were found, at a certain load for each of the samples, so that they can be easily compared. Figure 132 presents such results. As can be observed, the samples with the lower carbon content appear to be more resistant to contact fatigue than their higher carbon counterparts. In addition, the 0.8%C sample austempered at a lower temperature (220°C) behaved worse in SCF in comparison to the same sample austempered at 250°C.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Microhardness (HV)</th>
<th>Load (kN)</th>
<th>Number of cycles</th>
<th>Radial cracks initiation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C-250</td>
<td>695</td>
<td>15</td>
<td>5000</td>
<td>YES, RADIAL CRACKING</td>
</tr>
<tr>
<td></td>
<td></td>
<td>11</td>
<td>15000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>20000</td>
<td></td>
</tr>
<tr>
<td>08C-220</td>
<td>659</td>
<td>15</td>
<td>10000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>11</td>
<td>15000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>30000</td>
<td></td>
</tr>
<tr>
<td>08C-250</td>
<td>643</td>
<td>15</td>
<td>15000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>11</td>
<td>30000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>150000</td>
<td></td>
</tr>
<tr>
<td>06Nb-250</td>
<td>603</td>
<td>15</td>
<td>20000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>11</td>
<td>40000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>200000</td>
<td></td>
</tr>
<tr>
<td>06C-250</td>
<td>589</td>
<td>15</td>
<td>30000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>11</td>
<td>50000</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>9</td>
<td>300000</td>
<td></td>
</tr>
<tr>
<td>Bainitic Ref.</td>
<td>350</td>
<td>15</td>
<td>1800000</td>
<td>NO CRACKING FOUND</td>
</tr>
<tr>
<td>Pearlitic Ref.</td>
<td>247</td>
<td>15</td>
<td>1800000</td>
<td></td>
</tr>
</tbody>
</table>

Table 47: Cycles to cracking for the NANOBAIN grades and the two reference samples
The general behavior has been that the samples with higher hardness have behaved worse in SCF. It is known for traditional fatigue that higher yield strength materials (and therefore higher hardness) behave better at the same stress levels than other materials with lower yield strength. It is necessary to remember the nature of the test and that the results shown express Load vs. Cycles, not Stress. Since all the samples have different hardn esses, the stresses will not be the same for each of them because the load amplitude has been kept at 3 constant levels in every test. It has been observed that, for lower hardness samples, the radius of the indent left after the test is smaller in the higher hardness materials. This would mean that the stresses these materials are subjected to are higher than in the materials with lower hardness. Obviously, the stresses are high enough to cause the materials to fail at very low number of cycles. In view of this, before any conclusions are made with respect to the behaviour of these samples in SCF, a more in depth analysis of stresses will be done. It remains quite difficult to properly evaluate the behaviour of the materials based only on applied loads.

In Figure 133 it is possible to observe a plot of the cycles to failure with respect to surface hardness. As was mentioned above, the samples with the lower hardnes ses have a much better fatigue behaviour at the same applied load.

![Figure 132: Cycles before failure for each of the nanobainitic steels tested](image)

**Comparison with previous work at LTU**

Since the reference samples had not been tested at the moment of delivery of this report, another point of comparison has been provided so that the nanobain steels can be studied further at this stage. Table 48 presents the results for other various steels tested at 15 kN in the same SCF machine. Their hardness values have also been provided to ensure a better comparison.
As can be observed from the values in Table 48, all the previously studied steels either match or surpass the nanobainitic grades under study in terms of cycles to failure. It is of utmost importance to consider that the steel grades tested so far have been lab casts and, therefore, they have a considerable content of inclusions which are not normally present in industrial steels (which is the case for steels for which the comparison data that has been provided). Among other things the fatigue life of a material is sensitive of its defect content, decreasing rapidly when the defect content is higher. Since defects act as crack nucleation sites, then in order to get a better fatigue behavior the materials should possess as small amount of defects as possible.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Load (kN)</th>
<th>Number of cycles (fatigue life)</th>
<th>Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ausferritic 0.6%C (250°C)</td>
<td>15</td>
<td>600000</td>
<td>595-630</td>
</tr>
<tr>
<td>Induction hardened steel (240°C)</td>
<td>15</td>
<td>300000</td>
<td>600-630</td>
</tr>
<tr>
<td>Air hardened steel (160°C)</td>
<td>15</td>
<td>50000</td>
<td>600-700</td>
</tr>
<tr>
<td>Fully martensitic</td>
<td>15</td>
<td>30000</td>
<td>600-700</td>
</tr>
<tr>
<td>Previous works</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Actual work (Nanobainitic samples)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1C-250</td>
<td>15</td>
<td>5000</td>
<td>695</td>
</tr>
<tr>
<td>08C-220</td>
<td>15</td>
<td>10000</td>
<td>659</td>
</tr>
<tr>
<td>08C-250</td>
<td>15</td>
<td>15000</td>
<td>643</td>
</tr>
<tr>
<td>06CNb-250</td>
<td>15</td>
<td>20000</td>
<td>603</td>
</tr>
<tr>
<td>06C-250</td>
<td>15</td>
<td>30000</td>
<td>589</td>
</tr>
</tbody>
</table>

Table 48 Comparison of fatigue life of nanobain samples with previous work done at LTU
In Figure 134 it is possible to observe the fracture surface of one of the 06CNb-250 samples that has failed completely during one of the SCF tests. As can be seen the cracking occurred through a zone of the material that had a high content of inclusions that were as large as 100 microns. This is evidence that the steels tested so far have an amount of inclusions that will definitely influence the results obtained for the fatigue life.

Figure 134: SEM images of the inclusions in the fracture surface of the 06CNb-250 sample

Conclusions
The pearlitic and bainitic steels did not present any cracks after a very high number of cycles (1,800,000) at under the maximum load used in these tests (15KN). Meanwhile, the nanobainitic steels, which are far harder than the conventional bainitic steels, all presented cracking at a considerably low amount of cycles (5000). It was also possible to identify that, apparently, the SCF resistance of the nanobainitic steels was inversely proportional to their hardness. It is the authors’ speculation that this behavior is related to the fact that, at lower hardness values, the stress is much lower due to the higher contact area of the indenter. In consequence, if the stress is sufficiently low, the fatigue life will be longer.

In regards to the comparison of the nanobainitic steels to other industrial steels, it was observed that the behavior of the nanobain steels studied so far, was worse than any of the steels studied in previous works at LTU under the same test conditions. However, evidence has been provided that since, currently, the tests have been done on lab casts, the amount of inclusions could be the reason for such a poor fatigue behavior of the nanobainitic steels.
APPENDIX O : INVESTIGATING THE DUCTILITY OF 1CSI

Results on the identification of the microstructural parameters controlling the ductility and the TRIP effect taking place during tensile test of selected isothermal treatments are discussed and presented in this section.

Materials and experimental procedure.

The NANOBAIN steel used for this study is the 1CSi (Table 49) casted by Gerdau-Sidenor and treated isothermally by Ascometal using salt bath. The austenitisation conditions and salt bath conditions are presented in Table 50.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Mo</th>
<th>Cr</th>
<th>Nb</th>
<th>Al</th>
<th>Cu</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.98</td>
<td>2.90</td>
<td>0.77</td>
<td>0.004</td>
<td>0.45</td>
<td>0.04</td>
<td>0.21</td>
<td>0.16</td>
<td>0.016</td>
<td>0.014</td>
<td></td>
</tr>
</tbody>
</table>

Table 49 : Chemical composition in wt%.

<table>
<thead>
<tr>
<th>Tγ (ºC)</th>
<th>tγ (h)</th>
<th>TISO/tISO (ºC/h)</th>
<th>TISO/tISO (ºC/h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CSi</td>
<td>950</td>
<td>1h</td>
<td>220/22</td>
</tr>
</tbody>
</table>

Table 50 : Heat-treatments conditions as used at Ascometal.

Tensile tests were performed by Bosch, the conditions of the test were room temperature and a strain rate of 0.004s⁻¹, samples had 5 mm diameter and gauge length of 14 mm. The results presented in Table 51 correspond to the average of five tensile tests.

<table>
<thead>
<tr>
<th>YS/ MPa</th>
<th>UTS/MPa</th>
<th>U. Elon./%</th>
<th>F. Elon./%</th>
<th>HV10</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CSi-220°C</td>
<td>1704±21</td>
<td>2287±19</td>
<td>7.4±1.7</td>
<td>664±9</td>
</tr>
<tr>
<td>1CSi-250°C</td>
<td>1698±33</td>
<td>2068±8</td>
<td>11.6±0.5</td>
<td>613±20</td>
</tr>
</tbody>
</table>


Engineering stress/strain curves were provided by Bosch in order to analyze the TRIP effect through the determination of the incremental work hardening exponent “n”, defined as d (lnσ)/ d(lnεp), where σ represents the flow curve in the region of uniform true plastic deformation, and k is the strength coefficient. Thus engineering stress-strain curves together with their corresponding n v.s. true plastic strain curves that will be presented later in this document, are a representative case of the five experiments preformed by condition.

Metallographic samples preparation and observation under optical and electron microscopy and X-ray analysis were performed according to the procedures described in previous reports. Hardness was measured as HV10 the results corresponding to an average of at least 3 values.

Microstructural Characterisation.

The microstructure of 1CSi steel transformed at 220 ºC and 250 ºC is bainitic consisting of a mixture of retained austenite and bainitic ferrite. A summary of the detailed characterization of the microstructures studied is presented in Table 52.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Vab</th>
<th>Vγ</th>
<th>Cab/wt.%</th>
<th>Cγ/wt.%</th>
<th>t/ nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CSi-220°C-TENS</td>
<td>0.64±0.02</td>
<td>0.36±0.01</td>
<td>0.08±0.06</td>
<td>1.22±0.06</td>
<td>28±2</td>
</tr>
<tr>
<td>1CSi-250°C-TENS</td>
<td>0.66±0.02</td>
<td>0.34±0.01</td>
<td>0.05±0.06</td>
<td>1.47±0.06</td>
<td>28±1</td>
</tr>
</tbody>
</table>

Table 52 : Quantitative data on microstructure. Vi and Ci stands for the fraction and C content of the phase i, where i could be, β = bainitic ferrite and γ = austenite. t stands for the plate thickness of bainitic ferrite.
A detailed characterisation of the microstructure in terms of ferrite plate thickness and retained austenite morphology distribution has been performed. Figure 135 shows that both treatments, 220 and 250 °C, result in approximately the same bainitic ferrite plate thickness mean value, 28 nm, but distribution reveals a narrower distribution in the case of the microstructure obtained after isothermal heat treatment at 220°C.

![Figure 135: Bainitic ferrite plate thickness distribution and average value.](image)

Differences in the reported UTS could be explained in terms of higher C content in ferrite 0.08 v.s 0.05 wt.% , at 220 and 250 °C respectively, and higher dislocation density at 220 °C. It is important to remember that dislocation density, consequence of the plastic relaxation of the shape change accompanying bainitic transformation, increases as the transformation temperature decreases [HKDB, 2001a].

In relation with austenite distribution is necessary to distinguish between thin films (Figure 136) and blocks (Figure 137). Results show that in the case of thin film both the average size and the distribution are very similar. The situation changes in the case of the blocky austenite, Figure 137, where it is clear that an increase in the isothermal temperature leads to coarser blocks of austenite and the distribution, therefore, has a longer and wider “tail” towards the bigger sizes.
Figure 136: Retained austenite thin film morphology distribution.

Figure 137: Retained austenite blocky morphology distribution.
Mechanical behaviour. TRIP effect?

Mechanical behavior of the 1CSi microstructures are summarized in Figure 138. In the case of the microstructure developed at 220 °C, after a rapid increase there is a tendency to increase up to the instability criteria which is never reached, explaining the fact that all the elongation is uniform. Retained austenite seems to be moderately stable leading to an effective TRIP effect taking place before necking. The behavior of the 1CSi -250°C is remarkable in that, after the initial rapid increase, there is a decrease up to 2.5% true plastic strain, followed by a clear increase in the hardening that leads to very high values of uniform elongation (11%) and even higher fracture elongation (21%).

Keeping in mind that both microstructures have approximately the same amount of retained austenite, 35 %, the behavior described made us think of a heterogeneous distribution of C in retained austenite providing with a wide range of levels of mechanical stabilities in austenite which is spread through an equally wide range of plastic deformations. This heterogeneous distributions is very likely to be linked to the size distribution of retained austenite shown in Figure 136 and Figure 137, i.e. typical blocks between sheaves of bainite, whitish features in optical micrographs no bigger than 3 μm, also as blocks but this time only observable under a scanning electron microscope, few microns in size, and finally as thin films between the bainitic ferrite plates.

![Figure 138](image_url)

**Figure 138**: Stress-strain and “n” curves from the tensile tests performed on 1CSI.

In order to try to confirm the previous affirmation, a detailed study of the microstructure, not only the original but also after deformation has been carried out.

Deformed microstructure.

With the aim to assess the evolution and influence of retained austenite during the deformation process, three cuts of the tested tensile samples were prepared according to scheme in Figure 139.
After standard metallographic sample preparation the microstructure does not seem any different to the original one (un-deformed), i.e retained austenite and bainitic ferrite. This is not surprising as under the microscope any austenite that would have transformed to martensite, as a consequence of the applied stress, is indistinguishable from retained austenite.

On the other hand when retained austenite is tracked at the different deformation stages, Figure 140, some important information can be extracted. At the fracture surface (FS) no evidence of austenite has been found, but just beneath it, zone T1, is possible to observe that not all the austenite has transformed to martensite, almost 15% of retained austenite remains untransformed. It is also noticeable that about 20% of retained austenite transforms, up to T1 zone, and still the ductility achieved in one and the other microstructure is very different.

Another interesting fact is that the remaining un-transformed austenite, up to T1 zone, remains within the limits of what is considered the percolation threshold. It has been suggested that with microstructures of the kind considered here, failure in a tensile test occurs when the retained austenite loses continuity [SKh, 2010] and that this percolation threshold is reached when the austenite fraction reaches about 10%. Or in other words, it seems then that the formation of hard, stress/strain–induced martensite can only be tolerated if the austenite maintains a continuous path through the test sample.

A detailed observation of the X-ray peaks profile at the different deformation stages, Figure 141, also reveals the following:
Austenite lattice parameter tends to increase as deformation increases, which is linked with the fact that austenite with lower C content transform first to martensite being the remaining austenite richer in C i.e. having a bigger lattice parameter.

Asymmetry and shifting of the ferrite peaks as deformation increases, is a direct consequence of the appearance of martensite. Martensite and ferrite share the same 2theta positions.

Is there TWIP effect assisting ductility?

This question arose after observing the remarkable recovery of strain hardening in the 1CSi 250ºC microstructure, which is very typical of TWIP steels.

According to the latest literature review, [SCu, 2010; ADu, 2008; GDi, 2010; JNa, 2010], enhancement of ductility could be attained by means of TWIP effect, Twining Induced Plasticity. The deformation mechanisms and mechanical properties of face-centered cubic (fcc) metals, austenite, are strongly related to their stacking fault energy (SFE) $\gamma^*$ [SCu, 2010], which is the most crucial nucleation parameter determining whether twinning, martensite transformation or dislocation glide alone will occur during deformation of the material.

The stacking fault energy (SFE) is defined as:

$$\gamma^* = 2 \rho \Delta G^{\gamma\rightarrow\epsilon} + 2\sigma$$

where $\Delta G^{\gamma\rightarrow\epsilon}$ is the molar Gibbs energy of the transformation, $\rho = 4/(a^2 N \sqrt{3})$ is the molar surface density, $a$ is the lattice parameter and $N$ the Avogadro number, and $\sigma$ is the surface energy of the interface $\gamma / \epsilon$, with a value between 8-9 mJ m$^{-2}$.

And TWIP effect is said to occur when SFE lies in the range

$$12-18 \text{ mJ mol}^{-1} < \gamma^* < 35-45 \text{ mJ mol}^{-1}$$

A first attempt was made to calculate the value of SFE following the pseudo empirical expressions found in the related literature [SCu, 2010; ADu, 2008; GDi, 2010; JNa, 2010], and it was concluded that, as those expressions where derived for TWIP steels, which have very different chemical compositions and ranges of alloying elements, the expressions were unsuitable for our case. It was decided then to use MTDATA to calculate the necessary free energy change for $\gamma$ to $\epsilon$ transformation, using the chemical composition of the retained austenite present at both conditions, 1CSi at 220 and 250ºC, and derived from X-ray experiments, Table 53. The lattice parameter necessary for the calculation of SFE was also obtained from previous X-ray experiments.
To make sure of the consistence of the calculation procedure, the SFE was also calculated for a typical TWIP steel (0.08C, 27Mn, 0.52 Si, 4.1 Al) from ref. [SCu, 2010], and the results thus obtained compared with those reported in the same publication, finding that the results were almost identical, see Table 54. The SFE values reported for retained austenite of the 1CSi-220 and 250ºC are one order of magnitude bigger than those of the TWIP steel, and completely out of the range where TWIP is expected to take place, as specified. Therefore, and according to the theory, no TWIP effect should be expected in the studied conditions for Nanobain steels.

<table>
<thead>
<tr>
<th></th>
<th>220ºC</th>
<th>250ºC</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>1.22</td>
<td>1.47</td>
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<tr>
<td>Si</td>
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<td>Mn</td>
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<td>Ni</td>
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<tr>
<td>Mo</td>
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<tr>
<td>Cr</td>
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<tr>
<td>Cu</td>
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<tr>
<td>Ti</td>
<td>0.004</td>
<td>0.004</td>
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Table 53: Chemical composition, wt.%, and lattice parameter, nm, of retained austenite derived from X-ray data, for the 1CSi 220 and 250ºC microstructures.

<table>
<thead>
<tr>
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<th>SFE mJ mol⁻¹ (x10³)</th>
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<tr>
<td>TWIP</td>
<td>0.046*</td>
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<tr>
<td>1CSi_250</td>
<td>0.82</td>
</tr>
<tr>
<td>1CSi_220</td>
<td>1.06</td>
</tr>
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</table>

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[MTDATA] MTDATA Phase Diagram Software, National Physical Laboratory (UK)


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This report summarises the work carried out over the duration of the project.

The aim of this project was to investigate the fatigue and wear properties of Si-rich nanostructured bainitic steels (NANOBAIN).

A first phase of the project consisted in the design of different possible alloy compositions, divided into a first series tailored for the manufacture of small components using gas quenching, and a second series for the manufacture of larger sections heat-treated in a salt bath. Kinetics and basic mechanical properties were investigated on laboratory casts. During this first part, an unprecedented combination of tensile strength and ductility was achieved on a newly designed composition (UTS 2.2GPa, total elongation > 20 %). Wear rates as measured in twin-disc tests were as little as 50 % of those achieved on standard high-hardness bainitic grades.

In a second phase, two industrial heats were produced on the basis of results achieved during the first part of the project. These were used to manufacture component demonstrators or actual components and test them in representative conditions. Thus, tests on a metal scrap shear were carried out on the 0.6 %C grade designed in this project and heat-treated at 280 °C. Results were promising although testing conditions make it difficult to achieve a direct comparison. METSO estimates that this could provide similar performance as largely more expensive alloys, thus bringing a 10–20 % economic improvement. Fatigue testing at Bosch also led to promising results, with fatigue performance of the 06C grades on a par with those of 100Cr6. In contrast, results achieved on 1CSi were relatively poor, possibly due to poor cleanliness of the steel.

Studies and reports