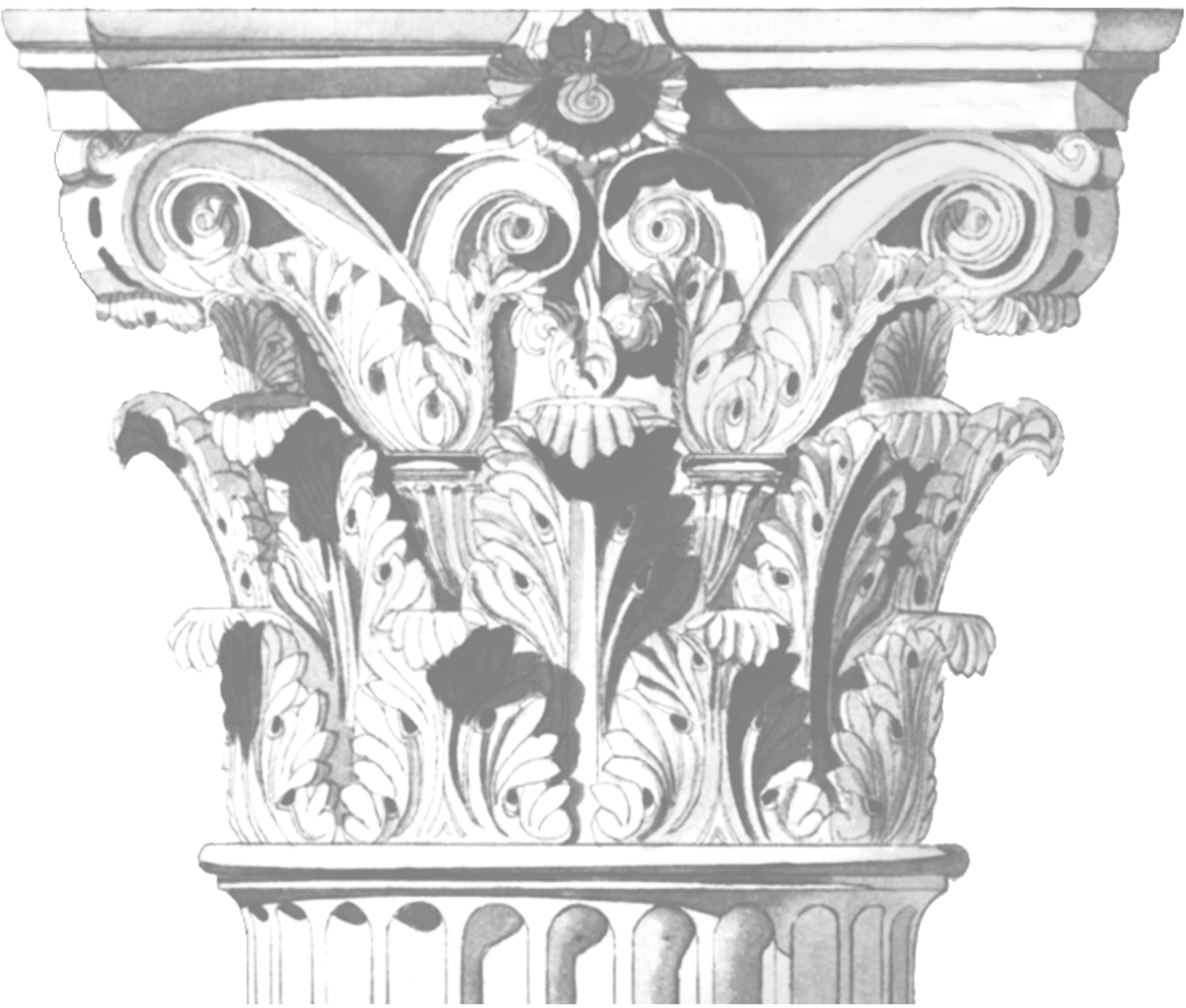
HOT DUCTILITY OF AUSTENITIC AND DUPLEX STAINLESS STEELS UNDER HOT ROLLING CONDITIONS

JUKKA KÖMI

Department of Mechanical Engineering,
University of Oulu

OULU 2001



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Academic Dissertation to be presented with the assent of the Faculty of Technology, University of Oulu, for public discussion in Raahensali (Auditorium L 10), Linnanmaa, on November 24th, 2001, at 12 noon.

OULUN YLIOPISTO, OULU 2001

Manuscript received 7 November 2001
Manuscript accepted 9 November 2001

Communicated by
Professor Veli Kujanpää
Doctor Juan H. Bianchi

ISBN 951-42-6560-2 (URL: http://herkules.oulu.fi//)

ALSO AVAILABLE IN PRINTED FORMAT
ISBN 951-42-6559-9
ISSN 0355-3213 (URL: http://herkules.oulu.fi/issn03553213/)

OULU UNIVERSITY PRESS
OULU 2001

Kömi, Jukka, Hot ductility of austenitic and duplex stainless steels under hot rolling conditions
Department of Mechanical Engineering, University of Oulu, P.O.Box 4200, FIN-90014 University of Oulu, Finland
2001
Oulu, Finland
(Manuscript received 7 November 2001)

Abstract

The effects of restoration and certain elements, nitrogen, sulphur, calcium and Misch metal, on the hot ductility of austenitic, high-alloyed austenitic and duplex stainless steels have been investigated by means of hot rolling, hot tensile, hot bending and stress relaxation tests. The results of these different testing methods indicated that hot rolling experiments using stepped specimens is the most effective way to investigate the relationship between the softening and cracking phenomena under hot rolling conditions. For as-cast, high-alloyed and duplex stainless steels with a low impurity level, the cracking tendency was observed to increase with increasing pass strain and temperature, being minimal for the small strain of 0.1. No cracking occurred in these steels when rolled in the wrought condition. It could be concluded that the cracking problems are only exhibited by the cast structure with the hot ductility of even partially recrystallised steel being perfectly adequate. However, the recrystallisation kinetics of the high-alloyed austenitic stainless steels, determined by stress relaxation and double-pass rolling tests, were found to be so slow that only partial softening can be expected to occur between roughing passes under normal rolling conditions. In the duplex steel, the restoration is fairly fast so that complete softening can occur within typical interpass times in hot rolling, while certain changes in the phase structure take place as well.

Sulphur was found to be an extremely harmful element in duplex stainless steel with regard to their hot ductility so that severe cracking can take place with sulphur content above 30 ppm. However, the effect of sulphur can be eliminated by reducing its content and by calcium or Misch metal treatments that significantly increase the number and decrease the average size of the inclusions. It seems that the desulphurisation capacity of an element is the most important property for assessing its usefulness in reducing the detrimental influence of sulphur.

The hot ductility of type 316L stainless steel determined by tensile tests was found to be better for nitrogen content of 0.05 wt-% than 0.02%, while in double-hit tensile tests the hot ductility values were identical. The mechanism whereby nitrogen affects hot ductility remains unclear but a retarding effect on static recrystallisation was observed.

Keywords: calcium, hot ductility, hot rolling, austenitic stainless steels, duplex stainless steels, recrystallisation, rolling test, tensile test, bending test, compression test, relaxation test, nitrogen, sulphur, Misch metal

Nothing is constant if not change

BUDDHA

Acknowledgements

This work was carried out in the Materials Engineering Laboratory, Department of Mechanical Engineering, University of Oulu and the Metallurgical Laboratory of Outokumpu Polarit Oy (currently AvestaPolarit Stainless) over the years 1991-2001, mostly as an avocation.

First of all, I am grateful to my supervisor Prof. L. Pentti Karjalainen for his valuable advice and countless critical discussions and suggestions during the course of the study. I would also like to thank my colleagues, the staff of the Research Centre at Rautaruukki Steel Oy, the Metallurgical Laboratory at Outokumpu Polarit Oy and the Materials Engineering Laboratory for their assistance in performing the experiments. I also wish to thank Dr. David Porter for correcting the English of the manuscript.

Finally I would like to express my warmest gratitude to my wife Pirkko and daughters Jenni, Hanna-Reeta and Henna-Riikka for they patience and unselfish support during this lengthy research.

Oulu, August 2001 Jukka Kömi

List of symbols

d	Delta ferrite
e	Strain
ε'	Strain rate (s^{-1})
g	Austenite
s_1	Initial flow stress during prestraining (N/mm^2)
σ_2	Flow stress immediately before unloading (N/mm^2)
σ_3	Initial flow stress during reloading (N/mm^2)
a, b, c	Constants (equations 2-4 in Introductory review and paper III)
A	Austenitic stainless steel
BC	Sum of crack lengths in hot impact bending test (mm)
BI	Cracking index in hot bending test
c	Laboratory cast (Table 1 in Introductory review and paper III)
CI	Cracking index in stepped hot rolling test
FA	Ferritic-austenitic stainless steel
FF	Ferrite fraction of ferritic-austenitic stainless steel
G	Gleeble test
h	Hot rolling test
HB	Hot bending test
HR	Conventional hot rolling test
HT	Hot tensile test
HV	Vickers microhardness (HV)
LCC	Sum of the lengths of the ten longest cracks in hot rolling test (mm)
P	Hot rolling force (N)
Q_{app}	Apparent activation energy of recrystallisation (J/mol)
Q_{def}	Activation energy for deformation (J/mol)
Q_{SRX}	Activation energy of static recrystallisation (J/mol)
r	Stress relaxation test
R	Hot rolling test
RA	Reduction of area in hot tensile test (%)
RC	Sum of crack lengths in hot rolling test (mm)
RI	Cracking index in conventional hot rolling test

S	Step hot rolling test
SA1, SA2	Super austenitic stainless steel (or high-alloyed stainless steel)
t_{50}	Time for 50% recrystallision (s)
t_{95}	Time for 95% recrystallision (s)
T	Temperature (K or °C as specified)
TE	Temperature at which the first cracks appear in hot rolled sheet (°C)
v	Elastic deflection of the rolls under loaded gap conditions (mm)
w	Wrought commercial material
W	Wedge hot rolling test
WC	Sum of crack lengths in wedge hot rolling test (mm)
Xb	Restoration index

List of original papers

I Kömi JI & Karjalainen LP (1993) Effect of hot working parameters on hot ductility of a ferritic-austenitic stainless steel. Innovation Stainless Steel, Florence, Italy, 11-14 Oct. 1993, pp. 2315-2320.

II Kömi JI & Karjalainen LP (1996) Effect of restoration kinetics on hot ductility of a ferritic-austenitic and super austenitic stainless steels. Proc. Int. Conf. on Stainless Steels, Düsseldorf, Germany, 1-3 June 1996, pp. 301-302.

III Kömi JI & Karjalainen LP. Effect of restoration on hot ductility of high-alloyed and duplex stainless steels. Accepted for publication in Mater. Sci and Technol.

IV Kömi JI, Kyröläinen AJ, Karjalainen LP & Suutala NJ (1991) Effect of sulphur, phosphorus and cerium on the hot workability of a ferritic-austenitic stainless steel. Proc. Int. Conf. on Stainless Steels, Chiba, Japan, 10-13 June 1991, pp. 807-814.

V Kömi JI, Karjalainen LP & Ruoppa R (1998) Hot ductility of nitrogen alloyed type 316L steel. Int. Conf. on High Nitrogen Steels, Espoo-Stockholm, Finland-Sweden, 24-28 May 1998[1].

The papers are referred to into text by their Roman numerals.

The planning of the experiments for papers I, II and III and the interpretation of the results were done by the author of this thesis and the writing of the manuscripts performed in collaboration with Prof. L. P. Karjalainen. The research plan for paper IV was prepared by Mr. A. J. Kyröläinen and Dr. N. J. Suutala. The author carried out the tests and analysed the results. The manuscript was written in co-operation with Prof. L. P. Karjalainen. The research plan for paper V was done by the present author and the tests were carried out in collaboration with Mr. R. Ruoppa. The manuscript was mainly on the responsibility of the present author.

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1. Submitted to the conference "High Nitrogen Steels 98", reviewed by two referees and accepted for printing and presentation as a poster and finally presented such as. Later, due to an unfortunate mistake during the compilation of the conference proceedings, however, the paper was misplaced and did not appear in the final publication. It will published in a magazine.

The novel experimental methods utilised and new ideas found by the present author and presented in the papers are as follows:

- Comparisons of the results of the several testing methods employed in this work made it possible to conclude that a hot rolling experiment with a stepped slab specimen is the most effective method for investigating the interaction between softening and cracking phenomena under hot rolling conditions. A quantitative measure of cracking tendency can be obtained by measuring the sum of the crack lengths and fractional softening can be estimated by using two-pass rolling tests, even though the stress relaxation method is evidently a more straightforward method to determine the recrystallisation kinetics.
- In high-alloyed austenitic and duplex stainless steels cracking problems only occur for the cast structure and the hot ductility of even slightly recrystallised material was adequate for further hot working. Cracking tendency was found to increase with increasing temperature and pass reduction. Therefore, a light first pass is recommended. A strain as small as 0.1 was found to be sufficient for improving the ductility.
- Microscopic examinations revealed the important roles of bulged austenite/ferrite interfaces on cracking in duplex stainless steels. Microanalyses and image analysis of inclusions indicated that the sulphur level as low as 30 ppm is detrimental to the hot ductility of duplex stainless steels while phosphorus has no detrimental effect. Calcium or Misch metal was found to be effective to prevent the harmful effects of sulphur.

Contents

1 Introduction

It is well known that poor hot ductility presents a problem in the manufacturing of high-alloyed and duplex stainless steels /1-5/. Even small amounts of impurities such as sulphur and phosphorus can impair the hot workability of the steel. Extensive studies have been performed on this topic, but still the deterioration mechanism is unclear. It is believed to be a consequence of reduced grain boundary cohesion causing voids and microcracks to be formed at the grain boundaries /1-3, 5, 6/.

The cracking problem has generally been solved using strict impurity and inclusion control /1-5/. Improving hot ductility by letting more restoration occur between the rolling passes is also a well-known method, but it is still not widely applied /3, 5, 7-11/. A reason for this is that the interaction between the restoration process and hot ductility is not yet clearly defined. In high-alloyed austenitic stainless steels the restoration kinetics are generally much slower than in type 304 or 316 steels and the recovery and recrystallisation can be retarded or even completely prevented at typical hot rolling temperatures /1-3, 7-11/. On the other hand, it has been observed that even though restoration rates are increased at high temperatures, the risk of cracking can still increase /2-5, 7-16/. In duplex stainless steels, restoration is accompanied by changes in the phase fractions and sizes, which makes the control of the process even more difficult /13-19/.

The hot ductility of steels has commonly been investigated employing hot tensile and bending tests /1-13/. Unfortunately, these methods can only provide qualitative results as regards hot workability in rolling. The main problem is the difference in the stress state in the hot tensile and bending tests compared to that in rolling, which may result in the creation of different cracking mechanisms. All these three testing methods have been used in the present work. Paper I deals with the effect of hot working parameters on hot ductility, employing hot impact, bending, tensile and rolling tests. In paper II and more extensively in paper III, the effect of restoration on hot ductility was investigated, in the former by tensile and rolling tests and in the latter by rolling tests. In bending and rolling tests, the hot ductility was characterised by counting the macroscopic cracks on the specimen surface with lengths greater than 0.5 mm. In tensile tests, the reduction of area was used as a measure of ductility.

The effects of impurities (S and P) and Ce alloying on the hot ductility of duplex stainless steel were studied in paper IV. Later, this investigation was extended by determining the effect of Ca and Misch metal treatments on hot ductility and by applying image analysis to the characterise the non-metallic inclusions.

The potential use of restoration for improving hot ductility is the main issue of the present work (papers II and III). In particular, the relationship between the softening and hot ductility phenomena was investigated using rolling experiments with stepped slabs. The softening was determined in two ways: by double-pass rolling tests and by the stress relaxation technique. The recrystallisation process has commonly been examined by quenching the deformed structure for subsequent metallographic inspection or by interrupted double-hit deformation tests to determine the degree of mechanical softening which is then related to the recrystallised fraction /1-5, 7-16/. At the University of Oulu, a novel technique, the stress relaxation method, has been developed and extensively utilised over several years to measure recrystallisation kinetics in hot-deformed austenite /20-22/. Hence, it was also employed here and the data on the recrystallisation kinetics were compared to that obtained from rolling tests (paper III). Furthermore, the influence of nitrogen content (≤ 0.05%) on the hot ductility and restoration of type 316L steel was also investigated (paper V).

Better understanding of material behaviour in hot rolling enables manufacturing of high-quality sheet or plate products of super austenitic and duplex stainless steels economically by utilising strict impurity control and controlled hot rolling schedules.

2 Experimental

Several as-cast and wrought stainless steels were investigated in this work: austenitic (paper V), high-alloyed or super austenitic (papers III, IV) and duplex (papers I, II, III, IV). The first groups of steels were used for investigating the effects of nitrogen alloying and the second and third groups for investigating the effects of restoration on ductility. The effects of impurities and inclusion modification were studied using duplex steel. The steels were of the types 17Cr-10Ni-2Mo (Polarit® 750), 20Cr-25Ni-4.5Mo-1.5Cu (Polarit® 774), 20Cr-20Ni-6Mo-0.2N (Polarit® 778) and 22Cr-5Ni-3Mo-N (Polarit® 809), referred to here as A1, A2, SA1, SA2 and FA, respectively. Their chemical compositions are given in Table 1.

Table 1. Average chemical compositions of the test materials, (wt-%).

Type and code (condition[1])	C	P	S	Cr	Ni	Mo	Al	Ca	N
austenitic, A1 (c, w)	0.023	0.024	0.0012	16.9	11.1	2.10	0.002	0.0011	0.019
austenitic, A2 (c, w)	0.022	0.029	0.0013	16.8	10.1	2.08	0.002	0.0010	0.046
super austenitic, SA1 (c)	0.018	0.022	0.0006	20.1	24.9	4.51	0.031	0.0034	0.055
super austenitic, SA1 (w)	0.016	0.021	0.0004	19.9	24.8	4.41	0.026	0.0037	0.046
super austenitic, SA2 (c)	0.016	0.012	0.0008	20.6	21.5	6.21	0.029	0.0029	0.189
super austenitic, SA2 (w)	0.014	0.021	0.0004	19.9	20.9	6.19	0.028	0.0022	0.190
ferritic austenitic, FA (c)	0.022	0.007-0.053	0.0003-0.0110	22.1	6.0	2.98	0.017-0.046	0.0003-0.0020	0.120
ferritic austenitic, FA (w)	0.015	0.022	0.0003	21.9	5.8	3.02	0.025	0.0013	0.171

[1] c: laboratory cast, w: wrought commercial material (rough-rolled).

Rolling tests were carried out in a pilot plant at AvestaPolarit Stainless that enabled the simulation of the rolling processes in roughing and finishing type mills. The pilot plant consists of a two-high mill housing with 450 mm diameter work rolls, two re-heating furnaces and thermal shields on both sides of the mill housing. The rolling sequences as well as gap settings are performed automatically while all rolling parameters are collected with a special data acquisition system at the rate of 50 Hz. The gap setting of the mill is fixed before the rolling passes because in-situ thickness control is not possible.

Before hot rolling, the slabs were heated in an air furnace to 1523 K, held for 30 min and cooled at the rate of 10 K/s to a pre-selected temperature. The hot working temperatures were in the range of 1223-1473 K with average steps of 50 K. After rolling, plates were cooled by a water spray at the rate of about 30 K/s. As a results of the constant rolling speed (0.5 m/s) and six different reductions the mean strain rate varied from 2 to 9 s^{-1}. The temperatures of the slabs were recorded (with inserted thermocouples) together with the mill forces.

In hot-rolling experiments stepped specimens were subjected to one or two passes. In the single-pass tests, six different mean true strains equal to 0.05, 0.11, 0.22, 0.36, 0.51 and 0.69 were obtained. The double-pass tests were performed to determine the degree of interpass softening and its influence on hot ductility, and therefore the specimens were subjected to a second pass with a true strain of 0.22 after various interval times.

Specimens for hot bending tests were heated at 1523 K for 10 min in an air furnace. The deformation temperatures were in the range of 1223-1473 K at average intervals of 50 K. After heating, the specimens were cooled to the deformation temperature and bent through 133° to a strain of 0.56 by subjecting them to an impact load with an energy of 750 J. Finally, they were water quenched.

Hot tensile tests were carried out at the University of Oulu using a MTS 810 testing machine with a radiant furnace and a Gleeble 1500 thermomechanical simulator with a resistance heating system. Specimens annealed at 1493 K were cooled to deformation temperature in the range 1123-1473 K at the rate of 2 K/s (MTS) or 20 K/s (Gleeble), strained to failure at the strain rates of 0.1-40 s^{-1} and finally air cooled to the ambient temperature. The tests were executed in the temperature range of 1123 to 1503 K. The double-hit tensile tests were carried out in the same way, but a second tensile strain was applied at various following the times after the first strain of 0.3.

The stress relaxation tests were carried out on a Gleeble 1500 thermomechanical simulator. Graphite foils were inserted between the specimen and the tungsten carbide compression anvils to reduce friction and tantalum foils to prevent sticking. They were heated at the rate of 10 K/s to 1493 K, held for 180 s, cooled to the deformation temperature at the rate of 10 K/s and held for 30 s to stabilise the temperature. Following this, the specimens were compressed to strains of 0.2 and 0.3 at a constant true strain rate of 1 s^{-1} and then the displacement (strain) was held constant while the decreasing compressive force was recorded as a function of time. The test is described in detail in Ref. 20.

The ductility of the hot-rolled steels was assessed by measuring the length of the cracks that appeared on the surfaces of the slabs during the tests. Cracks with lengths greater than 0.5 mm were measured under a stereo microscope. The sum of the lengths of individual cracks was used as a measure of the degree of cracking.

Microstructures were examined by means of optical and scanning electron microscopes (SEM). Ferrite fractions and the sizes and numbers of δ and γ phases in the duplex stainless steel were measured perpendicular to the rolling direction using an optical microscope. The nature of non-metallic inclusions was analysed using energy dispersive X-ray analysis and image analysis, with the aid of special software (SULKA) and statistical methods. A minimum of 250 inclusions was examined in each slab and the results were analysed in terms of size, shape and composition. The SULKA non-metallic inclusion program has been developed at AvestaPolarit Stainless /23/.

3 Results and discussion

3.1 Effect of restoration on hot ductility

3.1.1 Determining the degree of restoration in rolling

The effect of restoration on the hot ductility of as-cast steels under hot rolling conditions was discussed in papers II and III. The amount of restoration was measured using the hot rolling experiments with the stepped slab and by the stress relaxation technique. Rolling was performed using two passes with a certain pass interval to determine the degree of interpass softening. In Fig. 1, the hot rolling force at 1273 K is shown for the first and second passes with the interpass times of 10 and 60 s.

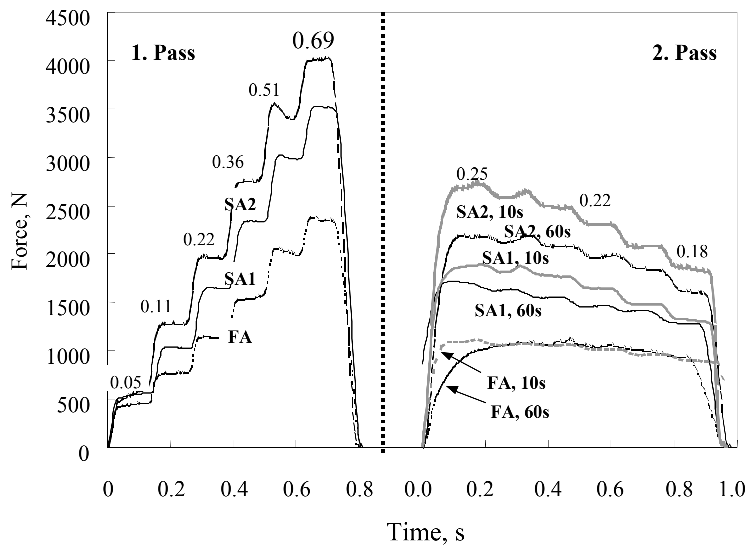


Fig. 1. Measured hot rolling force vs. time during the first and second passes at 1273 K. Interpass times are 10 and 60 s.

As seen, in the first pass the force increases in a stepped way due to the discontinuous increase in the reduction in the stepped slab (strain values are marked in the figure). The slab was still somewhat stepped after the first pass due to the constant gap settings and the elastic deflection of the rolls under loaded gap conditions (mill spring). Therefore, the strain varied slightly along the length of the slab in the second pass as well, as shown by the strain values given in the figure. Comparing the forces in the first and second passes, it can be observed that the rolling force clearly depends on the interpass time for the super austenitic steels. Hence, recrystallisation has taken place in the super austenitic steels for first-pass strains > 0.22 and in the duplex stainless steel for first-pass strains > 0.11**.** At lower strains, the force in the second pass seems to be mainly at a level corresponding to the accumulated reduction.

The rolling loads in the above figure only give a coarse measure of the degree of restoration. To analyse the phenomenon more thoroughly, stress strain curves were calculated. This analysis has to include the corrections necessary to compensate for the small slab thickness as well as the temperature differences that appeared between the passes. The thickness correction is a consequence of the constant gap setting and the mill spring. This phenomenon can be considered with the simple cubic equation (1):

$$P = -680v^3 + 2720v^2 - 1402v + 367 \tag{1}$$

where P is the rolling force (in N) and v the mill spring (in mm).

Normalising the examined temperature to the target temperature can compensate for temperature differences. This is necessary because none of the tests is made at exactly the same temperature and the second pass is always at a lower temperature than the first pass. The corrections are based on the assumption of a linear relationship between flow stress and temperature for the strains and strain rates used /3, 5, 29/. In this way, it is possible to normalise the measured flow stress to the target temperature useing a simple equation. In the calculations, six temperatures were used in the range of 1223-1473 K with 50 K steps. Based on these results, the dependencies of the mean flow stress on strain can be calculated for the interpass times of 10 and 60 s. From these curves the restoration index can be determined using the back-extrapolation method /5, 11/. Using this data, the time for 50% recrystallisation can be calculated.

The aim of the relaxation tests was to determine the quantitative time and temperature dependencies for recrystallisation. The results are displayed in Fig. 2, in which the time for the 50% recrystallisation is plotted as a function of the inverse of temperature. Relaxation test data for type 304 steel taken from Ref. 28 are included for comparison. From the figure, it can be seen that recrystallisation is markedly slower for both the SA steels compared to type 304, A or FA steels. The higher level of alloying is an obvious reason for the reduced recrystallisation rate in the SA steels. Especially Cr, N and Mo are known to decrease the recrystallisation rate /1, 3, 4, 7-12, 21/. It can also be assumed that the longer t_{50} times for SA2 are mainly a consequence of its higher Mo content compared to SA1. The faster restoration rate in the FA steel can be explained by the fine sizes of the δ and γ phases /13-19/.

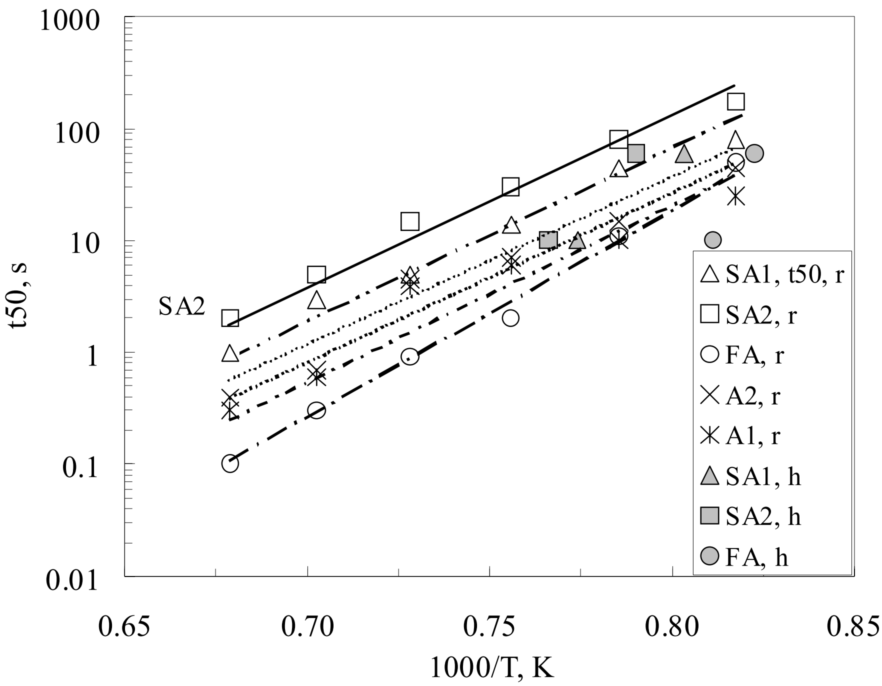


Fig. 2. Effect of temperature on the t_{50} determined from stress relaxation (ε = 0.20, ε' = 1 s^{-1}, marked by r) and hot rolling (ε = 0.22, ε' = 3.5 s^{-1}, marked by h). Data for A1 and A2 from paper V, for SA1 and SA2 from paper III, and for type 304 from Ref. 28.

However, to compare the softening fractions from these two methods, the different strain and strain rates as well as the strain distribution in the thickness direction in hot rolling must be taken into account. This has been discussed in papers II and III and a similar approach has been adopted in other papers /20-22/. The effect of strain rate on restoration is accounted for using the strain rate sensitivity exponent of -0.33 /11, 21/. It was also determined experimentally that at the surface layer of the slab the effective strain is about 26 % higher than at the centre. The strain exponent of -2.4 was used to account for the power of strain /10, 11, 22/. In the calculations, the redundant strain was also included /30, 31/. As a result, higher strain and strain rate and the strain distribution raise the static recrystallisation rate about 45 % comparing to that in the stress relaxation test conditions. Consequently, a reasonable consistency between the data from the both tests in Fig. 2 indicate that the laboratory rolling tests can also be used successfully to determine the softening kinetics, albeit not in a straightforward way but after several calculations.

3.1.2 Factors affecting hot ductility in rolling

The results in papers II-IV indicated that the rolling reduction and temperature are important factors that affect the risk of cracking of slabs in the cast conditions. The influence of these parameters on the cracking tendency during the first roughing pass can be combined using simple linear regression analysis. A cracking index (CI, in mm), defined as the sum of crack lengths was calculated for all results covering strains in the range 0.05-0.69, rolling temperatures from 1223 to 1473 K and strain rates from 2 to 9 s^{-1}. The combined effect of these variables can be represented by a simple equation for each steel:

$$CI = aT + b\varepsilon + c = 0.2T + 536\varepsilon - 334 \quad (SA1) \qquad (2)$$
$$CI = aT + b\varepsilon + c = 0.4T + 718\varepsilon - 571 \quad (SA2) \qquad (3)$$
$$CI = aT + b\varepsilon + c = 1.1T + 438\varepsilon - 1701 \quad (FA) \qquad (4)$$

where T (in K) is the roughing temperature and ε the strain. The accuracy of these equations is illustrated in Fig. 3. For practical hot rolling the results mean that, in the roughing stage, cracks nucleate and grow by mechanisms that are enhanced by high temperatures and strains. They also indicate that super austenitic steels are much more prone to cracking than the duplex steel. The influence of temperature is higher for duplex than for super austenitic, while the effect of strain is the opposite. At typical roughing temperatures ($T \geq 1373$ K) the avoidance of severe cracking requires strains to be as small as possible, at least below 0.2, especially for the super austenitic steels.

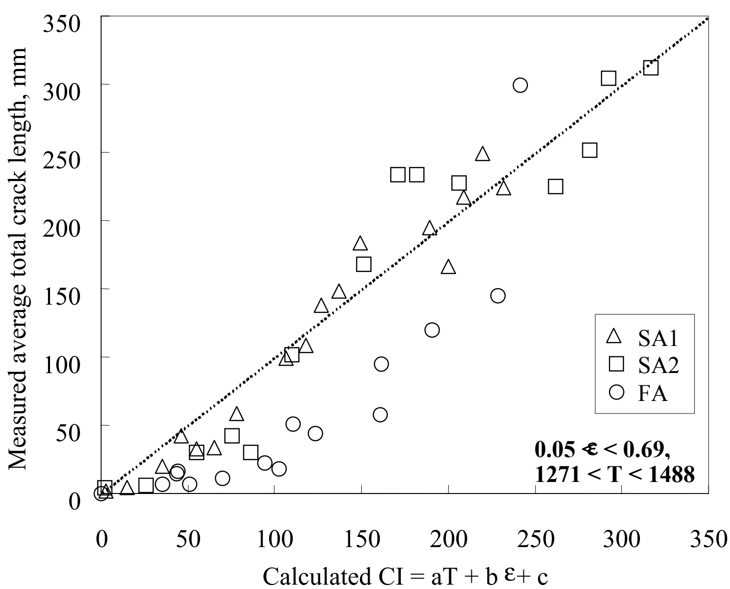
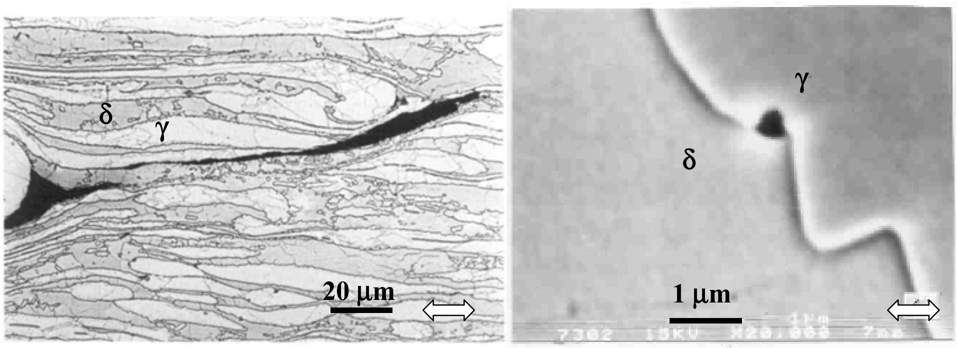


Fig. 3. Effects of temperature and strain on hot ductility (defined by CI). a, b and c are constants given in equations 2-4.

However, in paper I it was shown that according to bending and tensile test results FA steel shows a distinct low ductility trough located at around 1373 K at the strain rate of 40 s^{-1}, with the regime of low ductility extending to lower temperatures at lower strain rates. Similarly, hot torsion testing of an as-cast duplex steel /14/ and hot tensile tests of as-cast super austenitic steels /11/ have shown that ductility improves with increasing deformation temperature for a strain rate of 1 s^{-1}. Contrary to these, the present rolling tests clearly showed that cracking tendency increases with increasing temperature. This discrepancy may be a consequence of the different stress and strain distributions in rolling compared with tensile, bending or torsion tests. In duplex stainless steels the austenite and ferrite behave in different ways during deformation so that different stress states may induce different cracking mechanisms. It is also possible that the higher strain rate present in rolling only allows a slight dynamic restoration thereby enhancing grain boundary sliding and reducing ductility as suggested in paper I.

It has been shown in papers I and II that phase boundary bulging increases with increasing temperature and strain as does the frequency of cracking. It seems that the crack nucleates by bulging of the interface, when the strain and temperature are high enough. It may also be that the shape of the phase interface facilitates crack initiation between two bulges. It was found that micro-cracks always nucleate at incoherent δ-γ interfaces, as illustrated in Fig. 4. All this means that duplex stainless steels can be successfully rolled by reducing the hot working temperature below 1423 K, where the hot ductility is much higher (provided the pass strain is less than 0.1).



(a) (b)

Fig. 4. Typical microstructure and cracking (a) and the formation of the microcrack between two bulges (b) in a duplex stainless steel at the strain of 0.22 at 1373 K. Rolling direction marked ⇔.

3.1.3 Restoration and hot ductility of austenitic and duplex stainless steels

It was shown in papers I-III that strains as small as 0.1-0.2 are capable of creating such microstructural changes that markedly decrease the risk of cracking. These changes can be recrystallisation and consequent grain refinement in the super austenitic steels and, additionally, phase transformation in the duplex steel.

In order to reveal the relationship between fractional softening in the surface layer and the consequent improvement in ductility more clearly, the changes in the reduction of area and average total crack lengths are plotted as a function of fractional softening in Fig. 5. The change in the reduction of area means the difference in the RA values in tensile and double-hit tensile tests. In tensile test the specimen is strained continuously to failure, while in the double-hit tensile tests the specimen is pulled to a strain of 0.3 held for a time between 0 and 100 s and then strained to failure. The degree of softening was determined using stress relaxation testing after 0.3 compressive strain. For the hot-rolling data, the fractional softening is determined in single-pass tests with 0.36 strain and double-pass tests with 0.22+0.20 strain and interpass times of 10 and 60 s, see Figs. 1 and 3. The change in the average total crack length means the difference in the values obtained in the single- and double-pass tests.

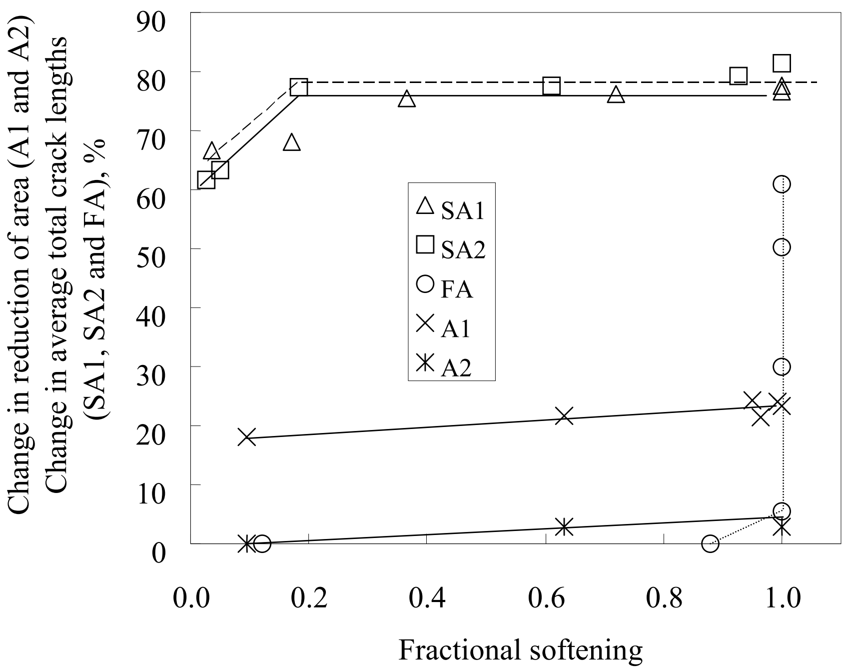


Fig. 5. The effect of statically recrystallised fraction on the improvement of hot ductility, defined as the change in the reduction of area or the change in the average total crack length in single and double-hit tests.

It can be seen in Fig. 5 that for both A and SA steels the change is quite independent of the fractional softening. Softening of even 5 % in SA steels leads to a very significant improvement in ductility. The results suggest that even a small amount of recrystallisation, i.e. a tiny number of grain nuclei formed along the grain boundaries can markedly inhibit cracking, as discussed in paper III. This observation can be explained if grain boundary sliding is the process that is responsible for loss of ductility during hot rolling since it will be effectively inhibited by the formation of new recrystallised grains at the old boundaries. In FA steel no significant cracking took place at low temperatures ($T < 1373$ K), so that no change could be expected, even though softening took place rapidly, clearly faster than in SA steels. Softening was complete at temperatures above 1273 K. At high temperatures the cracking tendency, however, increased, as can be concluded from Fig. 3 and equation 4. Therefore, at these temperatures, the improvement in the ductility cannot be connected with softening but requires certain other microstructural changes, as discussed in paper III.

In summary it can be concluded that laboratory hot rolling experiments employing a stepped slab specimen are an effective method for investigating the combined effect of restoration and hot ductility under hot rolling conditions. On the basis of a better understanding of this inter-relationship, cracking problems may be diminished enabling more economic production of strip with a better quality.

3.2 Effect of sulphur, calcium and Misch metal on hot ductility

The effects of impurities and inclusion modification by Ca or Misch metal on the hot ductility of duplex stainless steels were investigated using hot bending and hot rolling tests. The results are reported in paper IV. They indicate that hot ductility increases with decreasing sulphur content such that a considerably improved ductility is achived at the level of 30 ppm, although at around 1373 K a distinct low ductility regime still existed in hot bending tests and some cracking occurred in hot rolling tests. On the other hand, phosphorus content had no significant influence on hot ductility and concentrations up to 150 ppm did not increase the cracking susceptibility.

The addition of calcium or Misch metal was found to be very effective for improving hot ductility. In particular excellent hot ductility was achieved through the simultaneous addition of Ca or Misch metal with a low impurity content. Fig. 6 shows the regression relationships obtained in bending and rolling tests. Both results indicate that ductility is impaired quite rapidly by increasing impurity levels up to certain level, which may be explained by the saturation of grain boundary sites by impurity atoms.

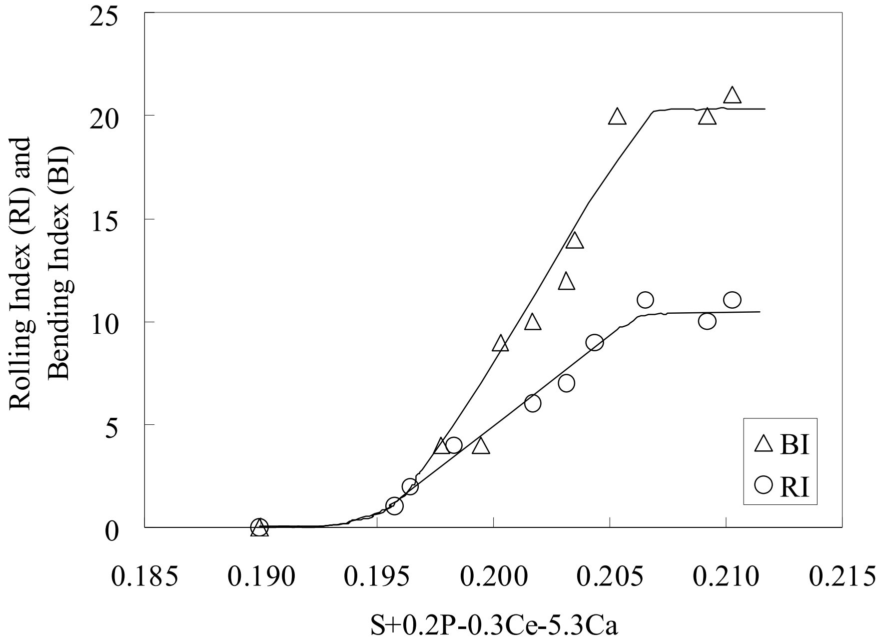


Fig. 6. Relationship between the cracking tendency and the composition factor S+0.2P-0.3Ce-5.3Ca in hot bending and hot rolling tests in FA steel.

The beneficial effect of the Misch metal and Ca treatment is based on the ability of the elements to bind the impurities and modify the shape and composition of the inclusions. Using thermochemical data for the formation of oxides and sulphides from liquid iron it can be shown that CaO or Ce_2O_3 as well as CaS, CeS or LaS will form from the melt /24-27/. Calcium is the most effective oxide former, but also the deoxidisation capacities of cerium and lanthanum are high. In fact, their desulphurisation capacities are higher than that of calcium /27/. Because all the steels are aluminium-killed so that oxygen is bound by aluminium, the desulphurisation capacity seems to be the most important factor in determining the effectiveness of added elements in reducing the harmful effect of sulphur. This conclusion is drawn from the results reported in paper IV and in Ref. 36.

It is known that in Ca treatment CaS is formed on the surface of the oxides that are typically of the type $12CaO{\bullet}7Al_2O_3$ /26, 27/. Correspondingly, after Misch metal treatment the core of the inclusions can be Ce_2O_3 or Al_2O_3-based oxides while the surface is typically LaS or CeS /27/. In analysing the effect of deoxidisation and desulphurisation processes on the type of inclusions, image and energy dispersive X-ray analysis were employed making use of the software SULKA /23/. In Fig. 7 the equivalent diameter distribution of non-metallic inclusions is illustrated. Only inclusions larger than 1.0 μm in diameter are taking into account. The equivalent diameter of >1 μm means the total density of inclusions (pcs/mm^2) bigger than 1 μm.

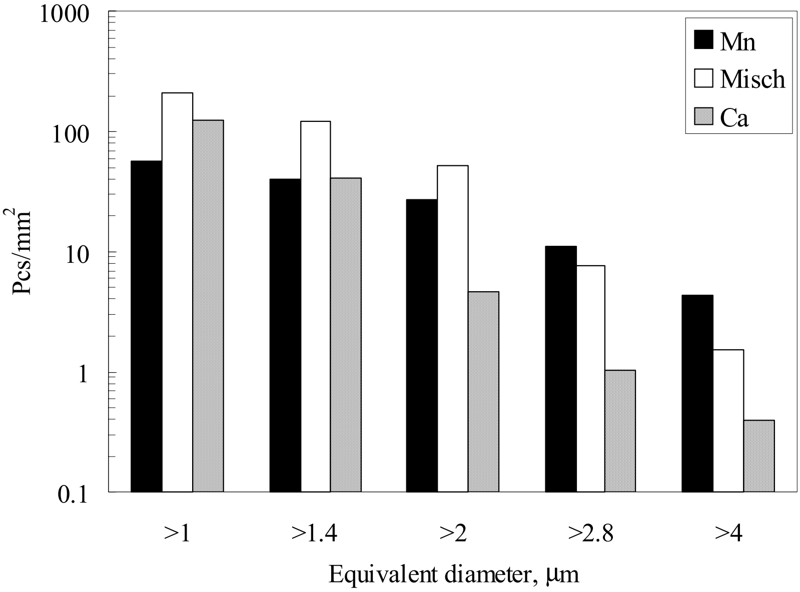


Fig. 7. Cumulative size distribution of the inclusions after treatment with Mn, Ca or Misch metal. All the steels are aluminium-killed (S = 0.001 and O = 0.003 wt-%).

The results in Fig. 7 show that the addition of calcium and rare earth metals clearly increases the number and decreases the average size of inclusions. It can also be seen that the effect of Misch metal is more significant than that of calcium. The changes in the number and size of inclusions are analysed in Fig. 8 where the contents of manganese, calcium and cerium/lanthanum are shown in mixed inclusions as a function sulphur content. In the figure, the stoichiometric lines for these elements are also drawn. If the contents of the appropriate elements after a given treatment are on or above the lines sulphides are formed effectively. This means that deoxidation was complete and there was enough manganese, calcium or cerium/lanthanum left to bind all sulphur.

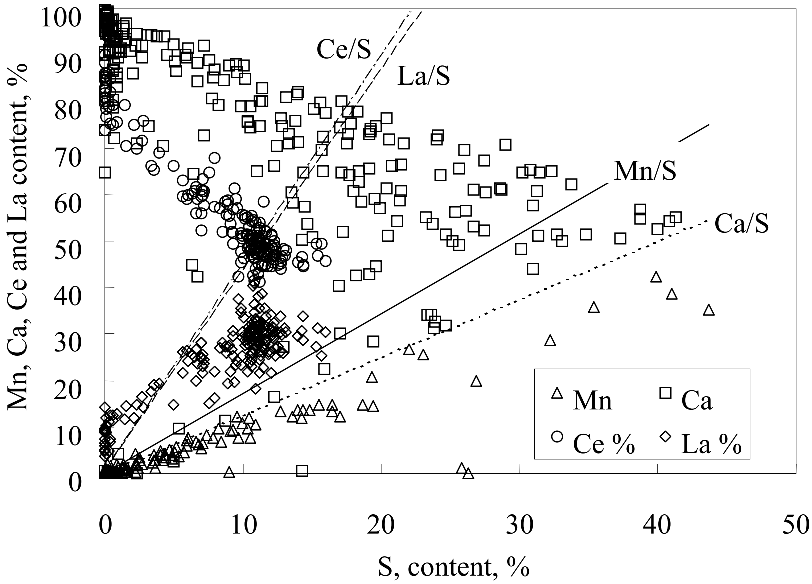


Fig. 8. Contents of Mn, Ca, Ce and La in inclusions as a function of S, Mn/S, Ca/S, Ce/S and La/S lines describe the stoichiometric relationships of these elements. All the steels are aluminium-killed (S = 0.001 and O = 0.003 wt-%).

It can be seen from Fig. 8 that the manganese and calcium contents in the inclusions increase with increasing sulphur content. On the other hand, the content of calcium is above the Ca/S stoichiometry line while the content of manganese is below the Mn/S line. This results from the fact that calcium treatment results in the reaction Ca with Al_2O_3 inclusions to form inclusion containing CaO while manganese forms MnS at the surface of different mixed oxides /26/. It can also be seen that manganese has a lower affinity for S than calcium, cerium or lanthanum, as also shown earlier /26, 27/. The influence of Misch metal is different. It is clearly seen that the content of cerium decreases and that of lanthanum increases with increasing sulphur content. This means that both cerium and lanthanum control the inclusion formation when the sulphur content of inclusions is high, but when it is low, the formation is controlled by cerium.

From the inclusion analysis illustrated in Figs. 6-8, one can conclude that the detrimental effect of sulphur on the hot ductility is prevented most effectively by calcium or Misch metal treatments. This means that the formation of CaS or CeS/LaS at surface of Al_2O_3-based oxides is the primary phenomenon. It is also clear that affinity of these elements for oxygen and sulphur is bigger than that of manganese causing the formation of calsium sulphides or cerium/lanthanum oxosulphides.

3.3 Effect of nitrogen on hot ductility

It is well known that a high nitrogen content decreases the hot workability of austenitic stainless steels /3, 9, 10, 37/. To elucidate the reasons for this, the effect of nitrogen (at low levels of 0.02 and 0.05 wt-%) and restoration on the hot ductility behaviour of type 316L austenitic stainless steel was investigated by hot tensile, double-hit tensile and stress relaxation test methods. The experiment are described and the results given in paper V. The hot ductility in hot tensile tests was found to be better at the higher nitrogen content. Similarly, Janzon et al. /37/ observed in hot tensile tests that nitrogen up to 0.08% improves the hot ductility. No significant effect on dynamic recrystallisation was found (at the strain rate of 1 s^{-1}) which indicates that dynamic restoration is a controlling mechanism. Therefore, the exact mechanism causing the improvement is not clear. Otherwise, no difference in the ductility could be observed in double-hit tensile tests. This could result from a retardation of recrystallisation kinetics due to an increased nitrogen content, revealed by the stress relaxation tests and shown Fig. 2.

4 Conclusions

The effect of restoration on the hot ductility of austenitic, super austenitic and ferritic-austenitic stainless steels has been investigated by means of hot rolling, bending, tensile and stress relaxation tests. Stress relaxation and double-pass hot rolling tests were used to determine the softening kinetics. Counting the number of cracks and measuring their lengths as well as measuring the reduction of area were used to assess the cracking tendency. Furthermore, in duplex stainless steel the effects of sulphur, phosphorous, calcium and Misch metal on hot ductility were studied. Inclusions were analysed using SEM-EDS and image analysis with the aid of a software package called SULKA. Microstructures and cracking mechanisms were also examined using optical and electron microscopes. The following conclusions can be drawn:

– Hot rolling using a stepped slab specimen is an effective method for investigating the interrelations between softening and cracking under hot rolling conditions. Hot bending and hot tensile tests can also provide data, but the results regarding the temperature dependence of the hot ductility differ from those of rolling tests, presumably due to the different stress states in the tests that induce different cracking mechanisms.
– Cracking problems occur in super austenitic and duplex stainless steels only for the cast structure. The hot ductility of even slightly recrystallised material is perfectly adequate for further hot deformation. The results suggest that a small amount of recrystallisation, i.e. just a few nuclei formed along grain boundaries can markedly reduce the risk of cracking. This, in turn, indicates that grain boundary sliding is the process that controls ductility during hot working.
– Cracks tend to nucleate in the first pass of the roughing stage and in order to avoid them, the reduction must be small. Cracking tendency increases with increasing strain and temperature. However, to ensure that static recrystallisation occurs to a sufficient extent before the second pass, the strain has to be at least 0.1 and the temperature above 1373 K, which lead to a fractional softening about 5% in the surface layer of the slab in super austenitic steels.
– In duplex stainless steel incoherent, bulged δ/γ-interphase boundaries play a significant role in cracking, both in crack nucleation and propagation. The length and number of the bulged interphase boundaries increased considerably at hot working temperatures above 1323 K.

- Sulphur is very detrimental to the hot ductility of duplex stainless steel at concentrations above 30 ppm. The harmful effects can be prevented most effectively by calcium or Misch metal treatments. Because in practice duplex steels are aluminium-killed, the affinity of the added elements for sulphur seems to be their most important property as regards the improvement of ductility, due to the formation of CaS or CeS/LaS at the surface of Al_2O_3 -type oxides.
- A phosphorus concentration up to 150 ppm has no effect on the hot ductility of duplex stainless steels.
- Increasing nitrogen from 0.02 to 0.05 wt-% improves the hot ductility of type 316 steel in tensile tests. The effect is not present in double-hit tensile tests, which may due to the retarded static recrystallisation at the higher nitrogen level. The reason for an improved ductility is not clear and calls for further investigation.

5 References

1. B. Mintz, M. Shaker and D. N. Crowther. Hot ductility of an austenitic and a ferritic stainless steel. Mater. Sci. Technol. 13(1997), pp. 243-249.
2. S. Tsuge. Effect of impurity and microalloying elements on the hot workability of duplex stainless steels. Proc. Int. Conf. on Stainless Steels, Chiba, Japan, 1991, pp. 799-806.
3. B. Ahlblom and R. Sandström R. Hot workability of stainless steels: influence of deformation parameters, microstructural components and restoration processes. Int. Met. Reviews 27(1982), pp. 1-27.
4. L. Myllykoski and N. Suutala. Effect of solidification mode on the hot ductility of austenitic stainless steels. Met. Technol. 10(1983), pp. 453-463.
5. D. J. Towle and T. Gladman. Recrystallization of austenitic stainless steels after hot rolling. Met. Sci. 13(1979), pp. 246-256.
6. T. G. Langdon. The role of grain boundaries in high temperature deformation. Mater. Sci. Eng. A166(1993), pp. 67-79.
7. S. Yamamoto, T. Sakiyama and C. Ouchi. Effect of alloying elements on recrystallisation kinetics after hot deformation in austenitic stainless steels. Trans. ISIJ 27(1987), pp. 446-452.
8. N. D. Ryan and H. J. McQueen. Comparison of static softening in 300 series stainless steels under iso- and anisothermal conditions. Mater. Sci. Technol. 7(1991), pp. 818-826.
9. A. Sandberg and R. Sandström. Recrystallisation of molybdenum- and nitrogen-alloyed austenitic stainless steels after hot working. Mater. Sci. Technol. 2(1986), pp. 917-925.
10. A. Sandberg and R. Sandström. Static recrystallisation and hot ductility of molybdenum- and nitrogen-alloyed austenitic stainless steels in associated with two- and multistep deformation. Mater. Sci. Technol. 2(1986), pp. 926-937.
11. R. A. Barbosa. Simulations of hot working of austenitic stainless steels. Ph.D. Thesis. Univ. of Sheffield, 1983.
12. T. Sakai. Microstructural development under dynamic recrystallisation of polycrystalline materials. Termomech. Processing of Steels. Eds. S. Yue and E. Essadiqui. Ottawa, Canada, 2000, pp. 47-62.

13. T. L. F. Müller. The deformation of duplex alloys. Ph.D. Thesis. Univ. of Sheffield, 1967.
14. A. Iza-Mendia, A. Piñol-Juez, J. J. Urcola and I. Gutiérrez. Microstructural and mechanical behaviour of duplex stainless steel under hot working conditions. Met. Trans. A, 29A(1998), pp. 1-11.
15. M. Barteri, E. Evangelista, M. Niewczas and L. Scoppio. Hot working and restoration mechanism in duplex stainless steels. Innovation Stainless Steel, Florence, Italy, 1993, pp. 3.285-3.290.
16. Y-H. Kim, S-C. Lee, C. G. Sun and Y-D. Lee. High temperature deformation behaviour and ductility of duplex stainless steel under hot working conditions. Stainless Steel '99, Science and Marketing, Sardinia, Italy, 1999, pp. 285-293.
17. E. Hornbogen and U. Köster, Recrystallisation of metallic materials. Ed. F. Haessner, Dr Riederer Verlag GmbH, Stuttgart, 1978, 159 p.
18. E. Hornbogen. Combined reactions. Met. Trans. A, 10A(1979), pp. 947- 972.
19. R. Mundt and H. Hoffmeister. Isothermal δ-γ transformation of ferritic-austenitic Fe-Cr-Ni alloys. Arch. Eisenhüttenwes. 54(1983), pp. 291-293.
20. L. P. Karjalainen. Stress relaxation method for the investigation of softening kinetics in hot deformed steels. Mater. Sci. Technol. 11(1995), pp. 557-565.
21. L. P. Karjalainen, J. A. Koskiniemi and X. D. Liu. Static recrystallisation in austenitic and ferritic stainless steels investigated by stress relaxation method. 37^{th} MWSP Conf., Hamilton, Ontario, Canada, 1995, pp. 861-869.
22. L. P. Karjalainen and J. Perttula. Characteristic of static and metadynamic recrystallisation and strain accumulation in hot-deformed austenite as revealed by the stress relaxation method. ISIJ Int. 36(1996), pp. 729-736.
23. T. Koskela. SULKA non-metallic inclusion software. Version 3.0. AvestaPolarit Stainless, Tornio, Finland, 2000.
24. Schlagenatlas. Düsseldorf, Germany 1981, Verlag Stahleisen M.B.H. 282 p.
25. ThermoCalc. Version K. Royal Institute of Technology, Div. of Computational Thermodynamics, Dept of Material Science and Engineering, Stockholm, 1995.
26. D-Z Lu, G. A. Irons and W-K Lu. Kinetics and mechanism of calcium absorption and inclusion modification of steel. Scaninject VI, Luleå, Sweden, 1992, p. 239-263.
27. L. Jiang and L. Holappa. Thermodynamic study on modification of non-metallic inclusion by Ca and Ce. Helsinki University of Technology, Dept. Materials Science and Rock Engineering, 1995, 33 p.
28. J. J. Välikangas, J. I. Kömi and L. P. Karjalainen. Characterisation of restoration kinetics of type 304 stainless steel. Modelling of steel microstructural evolution during thermomechanical treatment. ECSC Workshop. Brussels, Belgium, 1997, p. 203.
29. V. B. Ginzburg. Steel-rolling technology: theory and practice, New York, USA, 1989, pp. 343-394.
30. D. Q. Jin, I. V. Samarasekera and J. K. Brimacombe. Modelling inhomogeneous deformation during hot rolling of plain carbon steel. 38th MWPS Conf. Proc. ISS, Vol. XXXIV(1997). Iron and Steel Society/AIME, pp. 329-337.
31. F. Siciliano Jr., K. Minami, T. M. Maccagno and J. J. Jonas. Mathematical modelling of the mean flow stress, fractional softening and grain size during hot strip rolling of C-Mn steels. ISIJ Int. 36(1996), pp. 1500-1506.

32. C. Pedrix. Characteristics of plastic deformation of metals during hot working. Report CACA No. 7210 EA/311. The institute de Rechearches de Sidérurgie Francaise (IRSID), Saint Germainen-Laye, France, 1987.
33. K. Karhausen and R. Kopp. Application of FEM and elementary theory of plasticity to prediction of microstructure in hot rolling. 1st international conference on modelling of metal rolling processes. Eds. J. H. Beynon, P. Ingham, S.I. Mizban and K. Waterson. London, England, 1993, pp. 66-79.
34. M. Pietrzyk and J. G. Lenard. Modelling of microstructure during hot rolling of steels. 1st international conference on modelling of metal rolling processes. Eds. J. H. Beynon, P. Ingham, S.I. Mizban and K. Waterson. London, England, 1993, pp. 102-115.
35. W. Madej, J. Novak and R. Kuziak. Thermal-mechanical-microstructural model of hot rolling for austenitic steel plates. Modelling of Metal Rolling Processes 3. Ed. J. H. Beynon, M. T. Clark, P. Ingham, P. Kern and K. Waterson. London, England, 1999, pp. 42-49.
36. J. Kömi. Effect of impurities on the hot workability of ferritic-austenitic stainless steel. Master Thesis. University of Oulu, 1990 (in Finnish).
37. B. Janzon and A. Omsén. The Influence of Nitrogen on Hot Workability of Austenitic Stainless Steels. Clean Steel, Vol. 1. Eds. S. Brohult and E. Rudberg. Stockholm, Sweden, 1971, pp. 186-200.