High-chromium, low-nitrogen duplex stainless steels

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SYNOPSIS
An account is given of the production of three types of experimental high-chromium duplex stainless steel. The alloys had chromium contents of 22, 29, and 35 per cent. The partitioning of the main alloying elements between the austenite and ferrite phases was determined, and the effect of the alloying elements chromium, nickel, and molybdenum on the mechanical properties and corrosion resistance of the duplex stainless steels is summarized. The phase compositions of the different duplex stainless steels were measured by different techniques, and the values obtained were correlated. The cracking occasionally observed during the thermomechanical processing of these alloys was studied, and is explained as being the result of known embrittlement processes.

SAMEVATTING
Daar word verslag gedoen oor die produksie van drie soorte hoëchroom-dupleksvlekvryestaal met 'n chroominhoud van 22, 29 en 35 persent. Die verdeling van die hooflegeerelemente tussen die ousteniet- en ferrietfase is bepaal en daar word 'n opsomming gegee van die uitwerking van die legeerelemente-chroom, nickel en molibdeen op die meganiese eienskappe en korrosiebestandheid van die dupleksvlekvryestaal. Die fasesamestelling van die verskillende soorte vlekvryestaal is gemeet volgens verskillende tegnieke en die waardes wat verkry is, is gekorreleer. 'n Studie van die kraking wat soms tydens die termomeganiese verwerking van hierdie legerings waargeneem word, het getoon dat die kraking aan bekende verbrossingsprosesse toe te skryf is.

Introduction

While duplex stainless steels have been known and used on an industrial scale for about fifty years, they have attracted particular interest only in the last decade. This can be attributed to the successful selection of these alloys, especially after 1970, for service in demanding applications requiring both corrosion resistance and mechanical strength. In addition, there is now a much better understanding of the metallurgical features of these alloys[1]. Duplex stainless steels differ from the more widely known austenitic and ferritic stainless steels in that they consist of a mixture of the ferrite and austenite phases, usually in approximately equal proportions.

Conventional austenitic stainless steels (for example type 304) offer good general corrosion resistance, a high level of formability, good weldability, and excellent resistance to shock loading, but are of inherently low strength and are susceptible to various forms of selective corrosion, particularly in chloride environments. Ferritic steels (for example type 430) are significantly cheaper than the austenitic alloys. They possess excellent resistance to chloride-induced stress–corrosion cracking, but generally lack ductility and have limited general corrosion resistance. In addition, ferritic stainless steels are weldable only in thin gauges owing to serious embrittlement problems[2,3]. The advantages of austenitic and ferritic stainless steels can be combined in the form of duplex stainless steels, and these alloys have excellent resistance to both general corrosion and stress–corrosion cracking. A common example of a commercial duplex stainless steel is SAF2205, which is now manufactured in South Africa under licence[4]. All existing commercial duplex stainless

steels contain between 18 and 29 per cent chromium[5].

Nominal chemical compositions of the grades of stainless steel discussed above are given in Table I, along with the rands per tonne cost of the nickel they contain and a simplified indication of their corrosion resistance in South African minewaters. The nickel content of some of these alloys represents a significant component of their cost, and the potential cost savings offered by duplex stainless steels in this respect are significant.

The corrosion resistance of stainless steels is closely related to their chromium content and, in general, the higher the chromium content of these alloys, the better the corrosion resistance. Unfortunately, higher chromium contents in austenitic stainless steels must be balanced by a corresponding increase in the nickel content, and this causes a steep rise in cost. Alternatively, higher chromium contents in ferritic stainless steels confer an increased susceptibility to embrittlement. These difficulties are reduced in high-chromium duplex stainless steels since less nickel is required, and any potential embrittlement is confined to the ferrite phase.

The object of the research described in this paper was to determine whether it was possible to produce a duplex stainless steel containing 35 per cent chromium. Duplex stainless steels of 22 and 29 per cent chromium were also investigated for the purpose of comparison. The alloys were produced in a laboratory, and neither manganese or silicon was deliberately added to the compositions melted since it was considered that these elements would not have a significant effect on either the microstructure or the corrosion resistance. However, if the alloys were ever to be manufactured on an industrial scale, it would be necessary to include significant amounts of these elements for practical reasons. This paper describes the outcome of the work, as well as the thermomechanical processing route that was finally adopted for the manu-

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TABLE I

TYPICAL STAINLESS-STEEL ALLOYS AND THE COST OF THE NICKEL THEY CONTAIN (DECEMBER 1988 VALUES)

Alloy	Nominal composition	Structure	Nickel R/t	Corrosion resistance	
				General	Localized
3CR12*	Fe–12%Cr	Ferrite–martensite	–	Good	Adequate
Type 430	Fe–17%Cr	Ferrite	–	Good	Adequate
Type 304	Fe–18%Cr–9%Ni	Austenite	2457	Very good	Good
Alloy 20	Fe–29%Cr–20%Ni–3,25%Cu–2,25%Mo	Austenite	5460	Excellent	Excellent
SAF2205†	Fe–22%Cr–5,5%Ni–3%Mo–0,14%N	Ferrite–austenite	1505	Excellent	Excellent

* 3CR12 is a trademark of Middelburg Steel & Alloys Ltd
† SAF2205 is a trademark of Sandvik

TABLE II

CHEMICAL COMPOSITIONS OF THE DUPLEX STAINLESS STEELS

Alloy no.	Target composition	Composition*, % by mass						
		Cr	Ni	Mo	Si	O	N	C
377	Fe–22%Cr–9%Ni–3%Mo	22,0	9,07	2,81	0,070	0,021	0,006	0,03
378	Fe–22%Cr–9%Ni–3%Mo	22,1	9,20	2,89	0,025	0,042	0,006	0,03
379	Fe–22%Cr–9%Ni–3%Mo	22,4	9,14	2,82	0,030	0,050	0,004	0,03
380	Fe–22%Cr–9%Ni–3%Mo	22,4	9,24	2,92	0,030	0,037	0,005	0,02
413	Fe–29%Cr–14%Ni–3%Mo	28,5	14,4	2,70	0,020	0,080	0,002	<0,01
414	Fe–29%Cr–14%Ni–3%Mo	28,4	14,0	2,70	<0,02	0,080	0,002	<0,01
398	Fe–29%Cr–14%Ni–3%Mo	29,6	14,3	2,73	0,020	0,070	0,003	0,02
399	Fe–29%Cr–14%Ni–3%Mo	30,1	14,8	2,88	0,020	0,070	0,002	0,02
400	Fe–35%Cr–18%Ni–3%Mo	34,7	18,6	2,72	0,030	0,070	0,005	0,02
416	Fe–35%Cr–18%Ni–3%Mo	33,9	17,4	2,60	0,050	0,100	0,004	<0,01
448	Fe–35%Cr–18%Ni–3%Mo	35,0	18,8	2,50	0,030	0,,070	0,003	0,04
446	Fe–35%Cr–18%Ni–3%Mo	34,5	18,5	2,80	0,060	0,080	0,002	<0,01

* For all the alloys shown, the manganese content was less than 0,1 per cent and the sulphur and phosphorus contents less than 0,01 per cent

facture of these alloys. The corrosion resistance of these alloys will be described elsewhere.

Experimental

The alloys were prepared in a vacuum induction furnace under an argon atmosphere of low pressure. The compositions of the alloys, which are given in Table II, were calculated to give a 1:1 ratio of ferrite to austenite in the final product. All the alloys were hot-rolled from an original thickness of approximately 45 mm to a finished thickness of between 3 and 6 mm. Finally, each alloy was annealed at a suitable temperature to bring its microstructure to the desired ratio of ferrite to austenite. The hot rolling and annealing schedules are given in Table III.

The dual-phase structure of duplex alloys is a source of strength, and higher rolling loads or lighter passes than normal may be required[6]. The thermomechanical conditions chosen for the 22 per cent chromium alloy correspond closely to those used in industry for the commercial SAF2205 grade of duplex stainless steel (which also has a chromium content of 22 per cent), and no particular difficulties were experienced. However, it proved difficult to roll the 29 and 35 per cent chromium steels in the two-phase region owing to the occurrence of hot

TABLE III

HOT-ROLLING AND ANNEALING SCHEDULE FOR THE DUPLEX STAINLESS STEELS (ALL WATER-QUENCHED)

Alloy no.	Initial temp. before rolling* °C	Min. rolling temp. °C	No. of passes	Annealing temp. after rolling† °C	Final thickness mm
337	1100	1050	24	1060	3,10
378	1100	1050	24	1000	3,30
379	1100	1050	24	1060	3,15
380	1100	1050	24	1000	3,40
413	1300	1250	64	1050	5,25
414	1300	1250	64	1050	5,20
398	1300	1250	58	1050	5,10
399	1300	1250	58	1050	4,80
400	1400	1350	64	1050	5,10
416	1400	1350	64	1050	5,20
448	1400	1350	64	1070	5,00
446	1400	1350	62	1100	3,40

* The ingots were soaked for 1 hour at temperature prior to rolling
† The ingots were annealed for 1 hour after rolling

cracking, and these alloys had to be rolled at temperatures within the single-phase delta-ferrite field.

The annealing temperature necessary to convert each composition into a 50 per cent ferrite duplex alloy was determined experimentally. Examples of phase variation with temperature for the three types of alloy are given in Figs. 1 to 3. The percentage of austenite present was determined by both optical image analysis (IA) and X-ray diffraction (XRD). A typical XRD spectrum used in the calculation of the austenite volume fraction of a duplex stainless steel containing 22 per cent chromium is shown in Fig. 4. The main peaks due to the austenite and ferrite phases are clearly visible. Phase contrast for the IA was obtained by the use of potentiostatic etching in a 40 per cent sodium hydroxide solution (10 mol.dm^{-3}), a treatment that colours the ferrite phase yellow–brown and the austenite phase light blue.
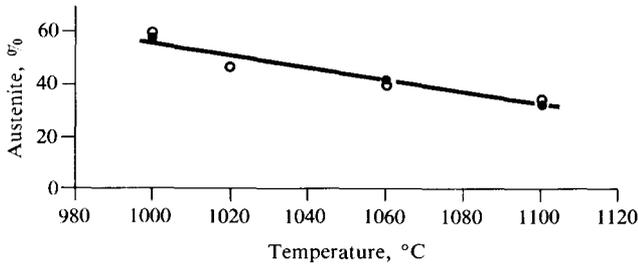
Fig. 1—Percentage austenite in duplex stainless steel of 22 per cent chromium as a function of temperature
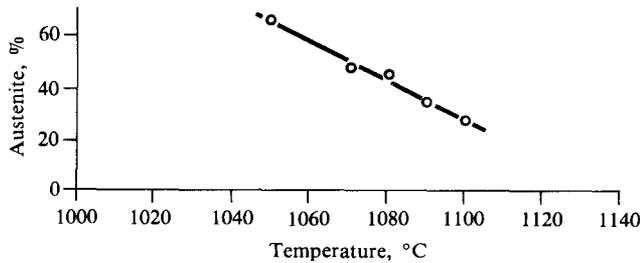

Fig. 2—Percentage austenite in duplex stainless steel of 29 per cent chromium as a function of temperature
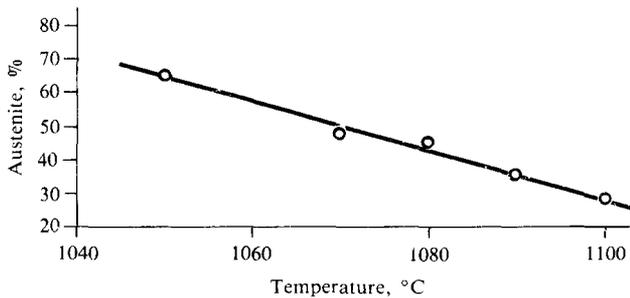

Fig. 3—Percentage austenite in duplex stainless steel of 35 per cent chromium as a function of temperature

Partitioning of the elements between the two phases was determined by the use of an electron microprobe (EPMA) and of the chemical-analysis facility on a scanning electron microscope (SEM).

Results

Microstructure

The austenite and ferrite phases formed well-defined alternating bands in the duplex stainless steel of 22 per
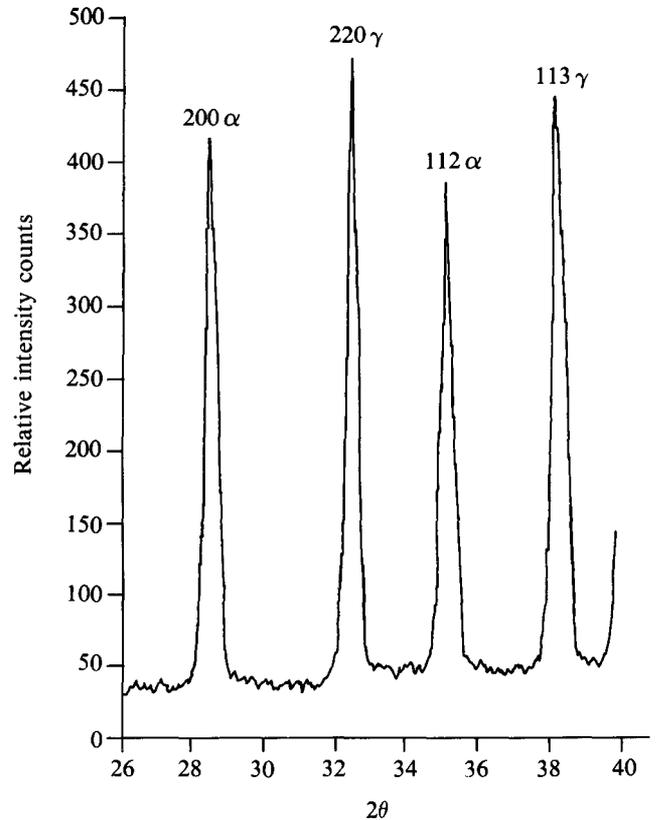

Fig. 4—An XRD spectrum of the austenite and ferrite phases present in a duplex stainless steel of 22 per cent chromium

cent chromium content (Fig. 5). This structure arises because the material was hot-worked within the two-phase field, with a consequent elongation of the two phases. The dark areas represent ferrite while the light areas in the photograph are austenite.
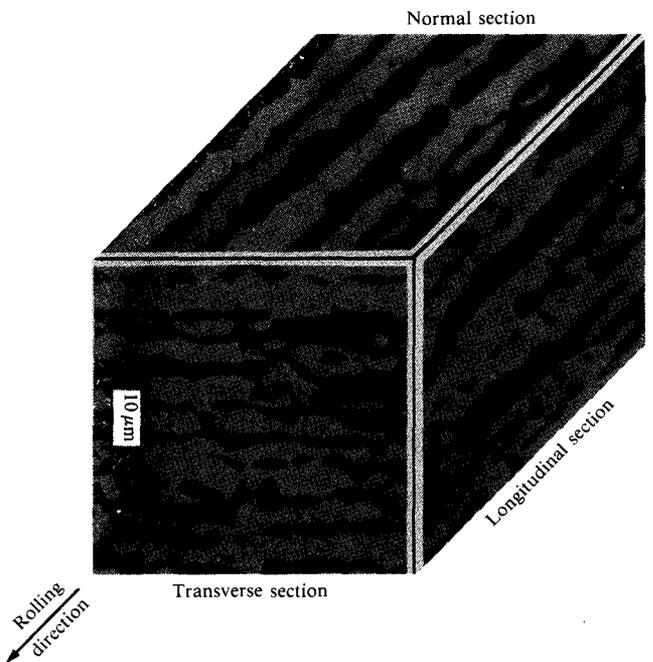

Fig. 5—Microstructure of a duplex stainless steel of 22 per cent chromium with alternating bands of austenite (light colour) and ferrite (dark colour)

The alloy of 29 per cent chromium was hot-worked at a temperature above the two-phase field. The austenite was transformed from ferrite during subsequent annealing within the two-phase field. This resulted in a less-pronounced banding of the structure (Fig. 6).

Normal section

Transverse section
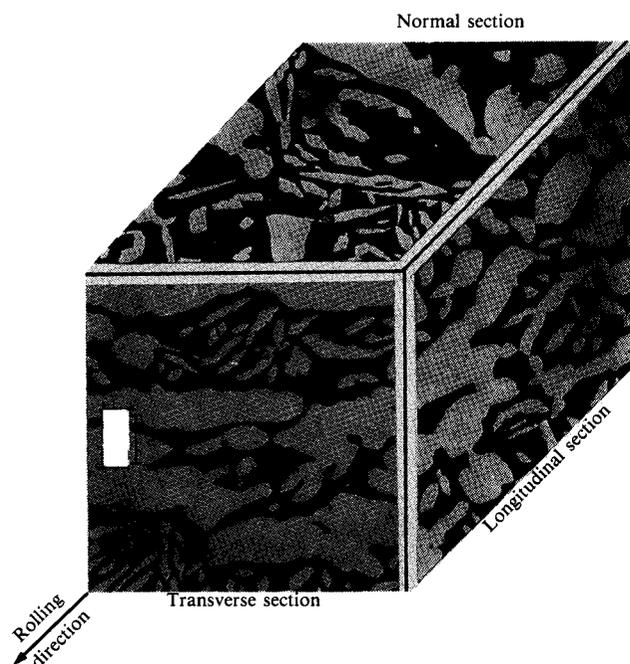
Rolling direction

Longitudinal section

Fig. 6—Microstructure of a duplex stainless steel of 29 per cent chromium with slight banding of austenite and ferrite phases in the longitudinal and transverse directions

The alloy of 35 per cent chromium showed (Fig. 7) no banding of the austenite and ferrite phases. The grain size of the delta ferrite was visible at low magnification because the delta-ferrite grain boundaries had been delineated by the austenite formed during the subsequent annealing.
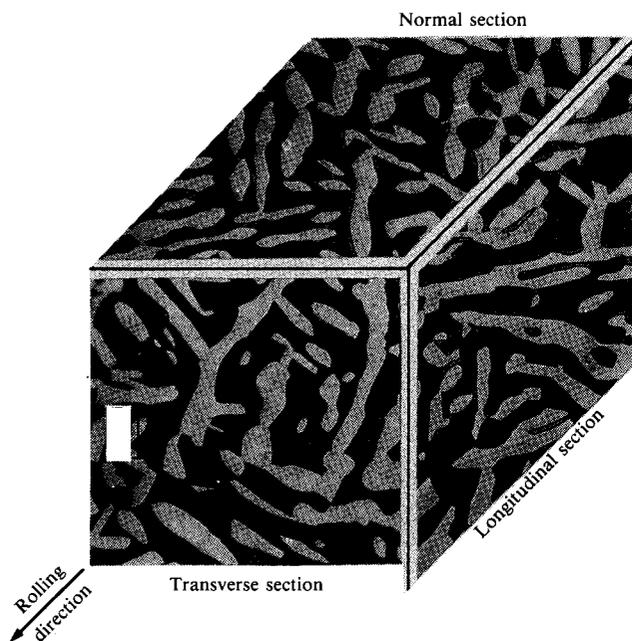
Normal section

Transverse section

Rolling direction

Longitudinal section

Fig. 7—Microstructure of a duplex stainless steel of 35 per cent chromium with a random distribution of austenite and ferrite grains

The ferrite grain size was also determined, and lay between 6 and 10 μm for the alloys.

Austenite-to-Ferrite Ratio

It is known that the mechanical properties of duplex stainless steel is greatly influenced by the ratio of austenite to ferrite within the alloys[7]. In particular, the hardness and yield strength are increased as the percentage of the ferrite phase increases while the elongation and impact energy decrease. The results of the phase determinations for the present alloys are summarized in Table IV.

TABLE IV
PHASE VOLUME FRACTIONS OF THE DUPLEX STAINLESS STEELS

Alloy type	Phase volume fractions measured by XRD %		Phase volume fractions measured by IA %	
	Ferrite	Austenite	Ferrite	Austenite
22% Cr	46,0	54,0 ± 3,4	46,6 ± 2,5	53,4
29% Cr	54,5	45,5 ± 3,5	54,6 ± 1,7	45,4
35% Cr	45,4	54,6 ± 2,0	58,0 ± 2,9	42,0

There was very good agreement between the results obtained by IA and those obtained by XRD for the 22 and 29 per cent chromium alloys, but not for the 35 per cent chromium alloys. The delta-ferrite grains in the latter were very large prior to cooling (3 to 4 mm), and this resulted in a preferred orientation in the subsequent duplex structure. The presence of such crystallographic texture is known to influence the intensity and clarity of the diffraction peaks, which was observed for this material. The results of the metallographic IA are thus more accurate for the 35 per cent chromium alloy.

Partitioning of Alloying Elements

It is well known that chromium and molybdenum are ferrite stabilizers, while nickel is an austenite stabilizer. It is also known that, in a duplex alloy, these alloying elements tend to be present in different concentrations in the ferrite and austenite phases[1]. The existence of this partitioning affects both the corrosion resistance and the mechanical properties of duplex stainless steels. The partitioning of the different elements in the present series of alloys was determined by both SEM and EPMA measurements. The results are given in Table V.

It can be seen that, notwithstanding the significant difference in chemical composition between the three types of alloy, the degree of partitioning of the elements between the two phases remained relatively constant.

Cracking during and after Hot-working

Hardness determinations indicated that the hardness of these duplex stainless steels increased with increasing chromium content (Fig. 8). This may be due to the effect of substitutional strengthening.

In addition, the duplex structure of these steels contributed significantly to their strength. As mentioned, it was found that the materials containing 29 and 35 per cent chromium were difficult to roll at temperatures within the two-phase field. Cracking of the slabs in this case is known to be a consequence of the differential

TABLE V

TABLE V
THE AVERAGE PARTITIONING OF ELEMENTS IN THE AUSTENITE AND FERRITE PHASES OF THE DUPLEX STAINLESS STEELS

Alloy type	Method	Ferrite phase			Austenite phase		
		%Cr	%Ni	%Mo	%Cr	%Ni	%Mo
Fe–22%Cr–9%Ni–3%Mo	EDS	26,3	6,1	3,9	19,6	11,0	2,4
	WDS	24,6	6,0	3,2	18,3	10,8	1,8
Fe–29%Cr–14%Ni–3%Mo	EDS	35,8	9,3	3,6	24,5	18,2	2,2
	WDS	34,8	9,2	3,6	23,6	18,1	2,0
Fe–35%Cr–18%Ni–3%Mo	EDS	40,9	12,3	3,6	27,9	23,2	2,1
	WDS	41,2	12,4	2,9	27,7	23,4	1,6

EDS Measured by SEM
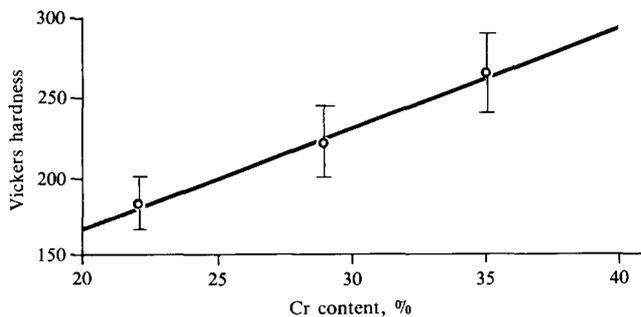WDS Measured by electron microprobe

Fig. 8—Hardness versus chromium content in duplex stainless steels

yielding characteristics of the two phases. In particular, the ferrite phase has a considerably lower strength than the austenite phase at elevated temperature, and thus deformation during hot-working tends to be concentrated in the ferrite grains[5]. The resultant stress concentration at the austenite–ferrite interfaces may cause crack initiation and propagation during rolling.

This problem was solved for the alloys of higher chromium content by rolling them at temperatures above the two-phase field. The higher temperatures, however, allowed a variety of new embrittling mechanisms to become possible.

The first of these was 'burning' of the ingot or plate during soaking. 'Burning' occurs when an alloy is heated to such a high temperature that either incipient melting occurs at its grain boundaries or rapid intergranular oxidation occurs. In either case, the alloy almost falls apart along the grain boundaries when plastically deformed at elevated temperature.

An example of incipient melting caused by 'burning' is shown in Fig. 9. The characteristic interdendritic morphology can be clearly seen. EDS analysis on the SEM indicated that the surface of the cracks was liberally coated with Cr_2O_3. Such interdendritic cracks normally form on the surface of ingots, especially at their edges during rolling. Once formed, the cracks are propagated further into the material by hot tearing, chiefly along a transgranular path.

'Burning' can be avoided by ensuring that the temperature of the ingot or plate does not rise too high during soaking or reheating. However, when the interpass temperature is reduced, another type of embrittlement

Fig. 9—Interdendritic morphology of a crack at the edge of a 'burnt' ingot of a duplex alloy (35 per cent chromium)

takes place. Many highly alloyed steels are susceptible to the formation of a brittle, intergranular sigma phase. The temperature up to which the sigma phase is stable, and the speed at which it forms by nucleation and growth from the parent phases, are greatly enhanced by alloying elements such as nickel, chromium, and especially, molybdenum[2,5]. XRD studies at elevated temperatures revealed that the sigma phase was stable in the present 35 per cent chromium alloys at temperatures up to 1090°C. Since the temperature of an ingot drops during rolling, it was necessary to hot work at temperatures substantially above 1090°C.

Fig. 10 shows the fracture surface of a 35 per cent chromium alloy that cracked when heated from 20 to about 1000°C. The mechanism of crack propagation is

brittle cleavage within the ferrite phase and ductile microvoid coalescence within the interleaved austenite phase. Metallographic examination of this alloy by use of potentiostatic etching revealed the presence of the sigma phase within the ferrite grains. This alloy had been annealed at 1070°C, and subsequent high-temperature XRD revealed that the sigma phase had formed in this alloy at temperatures up to 1090°C. The primary cause of the reduced toughness of the ferrite was the presence of the sigma phase.
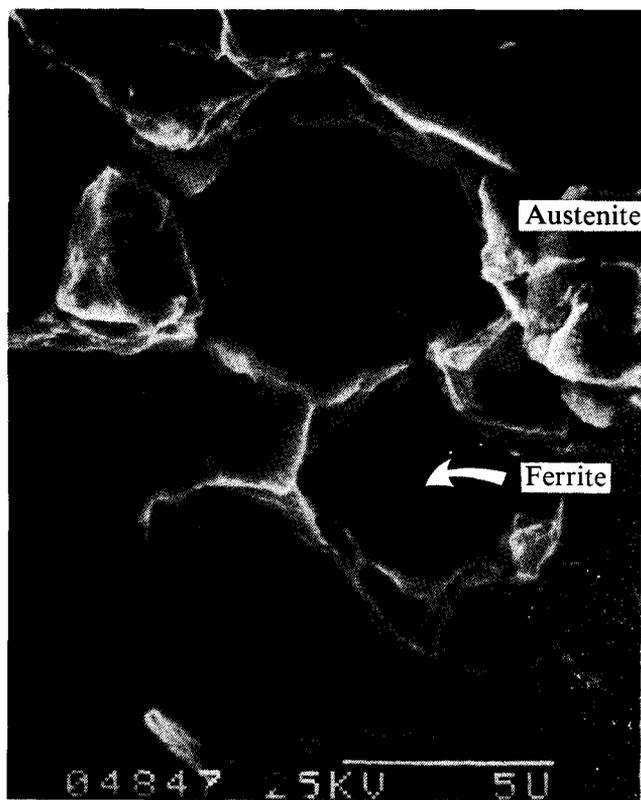

Fig. 10—Brittle crack propagation through the ferrite phase of a duplex stainless steel (35 per cent chromium)

Yet another type of embrittlement that may occur in these alloys is known as high-temperature embrittlement. This phenomenon is related to the carbon and nitrogen levels in the ferrite and may occur[2] in alloys heated above about 1000°C. For the present series of alloys with their low carbon and nitrogen contents, it can be expected that slow cooling from high temperatures through the critical 500 to 700°C range would lead to precipitation of the little carbon and nitrogen actually present as chromium carbides and nitrides, thus reducing the ductility of the alloy. In contrast, quenching from 1000°C would result in the interstitial elements being retained in solid solution with no deleterious effect. The situation would be reversed for higher interstitial contents and, for such alloys, rapid cooling from the solution annealing temperature is undesirable and slow cooling is preferred[2].

Besides the high-temperature embrittlement processes, a phenomenon known as 475°C embrittlement occurs in many ferritic and duplex stainless-steel alloys. This process takes place when the alloy is held at temperatures between 400 and 500°C, and its duration varies from

thousands of hours (for chromium contents of 15 per cent) to several minutes (for chromium contents of over 25 per cent). Although the occurrence of this phenomenon in ferritic stainless steels is well known, it should be noted that it can also occur in duplex stainless steels, even those containing as little as 15 per cent ferrite or 19 per cent chromium[2,4,5,8–10]. The embrittlement is caused by the spinoidal decomposition of the chromium-rich ferrite phase. An alloy that has been severely embrittled by this process may shatter when dropped, or when subsequently reheated for further processing.

Discussion
The Role of Chromium, Nickel, and Molybdenum
All stainless steels derive their passivity from alloying with chromium. Such alloying expands the passive potential range by moving the primary passivation potential, E_{pp}, in the active direction[1,5]. It also reduces the passive current density, i_{pass}. Chromium is beneficial in increasing the resistance to pitting and crevice corrosion in chloride environments because it raises the pitting potential, E_p, to more noble potentials, thus expanding the passive potential range[1]. However, the significant advantages of an increased chromium content are partially offset by the problems of embrittlement previously discussed.

Molybdenum has a strong beneficial influence on the general corrosion resistance of an alloy, as well as on the resistance to pitting and crevice corrosion in chloride media. This can be ascribed to the fact that additions of molybdenum extend the passive potential range by moving the pitting potential in the more noble direction[1]. They also lower the critical current density, i_{crit}, at the onset of passivation, thus enhancing the ease with which passivation can be established. However, additions of molybdenum widen the sigma phase field and can extend[5] it to temperatures in excess of 1000°C. Therefore, the maximum amount of molybdenum that can be added to duplex stainless steels is restricted to around 4 per cent.

Additions of nickel to duplex stainless steel have been reported to increase resistance to both crevice corrosion[11] and pitting corrosion[12]. Like chromium, nickel moves the pitting potential in a more noble direction, thereby extending the passive potential range. It also reduces the passive current density. However, if the nickel contents are significantly in excess of the optimum for pitting resistance so that the austenite content increases markedly above 50 per cent, the residual, more highly alloyed ferrite, as a result of partitioning, will be more readily transformed to the brittle sigma phase at temperatures in the range 650 to 950°C. Conversely, if the nickel content is reduced below the optimum level for pitting resistance, leading to high ferrite contents, low toughness will result because the delta ferrite that forms immediately on solidification tends to have low ductility[13]. It therefore seems probable that the main role of nickel is to control the microstructure, rather than to modify the corrosion resistance *per se*.

Hot Workability and Cracking
As a rule, duplex stainless steels have poor hot-workability[5], which is a consequence of their microstructure.

The preferred ferrite level is between 40 and 60 per cent because higher or lower ferrite contents result in somewhat poorer mechanical properties[5,7,14], as discussed in preceding sections. Although superplasticity can occur during the hot-working of duplex alloys[5,15], this was not found in the present alloys.

Ductility depends on the composition and heat treatment of steel. The low impact properties and ductility of nitrogen-free alloys are particularly striking in heat-treated and thick sections[13]. As austenite is tougher than ferrite, the large difference in hardness between the two phases at the hot-working temperature will increase the risk of crack formation. It may therefore be advisable to hot-roll duplex stainless steels at the single-phase temperature to avoid cracking, as was found to be the case in the present work for the 29 and 35 per cent chromium alloys. However, the rolling temperature must be carefully chosen in order to avoid the other types of embrittlement discussed previously.

It is important to subject high-chromium duplex stainless steels to solution heat treatment (annealing) after melting, casting, and hot-working. This solution heat treatment not only adjusts the microstructure of alloys to the desired volume fraction ratio of austenite to ferrite, but also gives rise to a product that is substantially free of precipitated particles. This improves the corrosion resistance[16]. Depending on the carbon and nitrogen content, it can be advantageous if, after such solution heat treatment, alloys can be cooled in air rather than being rapidly cooled by quenching in oil or water. The solution heat treatment followed by rapid cooling may result in the creation of excessive stresses in the duplex steel. It is obviously advantageous for alloys to have adequate ductility, which would allow cooling without cracking, especially in large forged shafts and other heavy sections subject to large thermally induced stresses.

Prediction of Equilibrium High-temperature Phases

In a multicomponent system consisting of iron, chromium, nickel, and molybdenum, the primary solidification mode for different Cr_{eq}/Ni_{eq} ratios can be predicted[5,17-19]. If (in mass percentages)

$$Cr_{eq} = Cr + 1,4\,Mo + 1,5\,Si + 3\,Ti \quad\text{.............} \quad (1)$$

$$Ni_{eq} = Ni + 0,3\,Mn + Cu + 22\,C + 14\,N, \quad\text{......} \quad (2)$$

then the solidification is primary austenite at Cr_{eq}/Ni_{eq} values of less than 1,5, while the solidification is primary ferrite if the Cr_{eq}/Ni_{eq} ratio is equal to or greater than 1,5. According to Hoffman[4], the optimum hot ductility for a duplex stainless steel of 22 per cent chromium is achieved at a Cr_{eq}/Ni_{eq} ratio of 1,5. In the present investigation, the Cr_{eq}/Ni_{eq} ratios for the duplex stainless steels of 22, 29, and 35 per cent chromium averaged 2,67, 2,23, and 2,03 respectively, indicating that all three types should have solidified as ferrite. The phase formed on solidification in the duplex stainless steels of 29 and 35 per cent chromium was primary ferrite, in agreement with the predictions by Suutala[18,19], but it was not clear which phase formed first when the 22 per cent alloys solidified.

Conclusions

(1) Duplex stainless steels containing as much as 35 per cent chromium can be produced with an austenite-to-ferrite phase ratio of 1:1. This ratio is considered to be the optimum for good corrosion resistance and mechanical properties.

(2) Quantitative proportions of the two phases present can be determined accurately by the use of either metallographic IA or XRD analysis.

(3) Partitioning of elements can be accurately determined by either SEM or EPMA techniques, and is significant in the high-chromium duplex stainless steels discussed here.

(4) Hot-working of duplex stainless steel containing 22 per cent chromium does not present a problem, even in the two-phase temperature range but, for duplex stainless steels of higher chromium content, it seems best to conduct hot-rolling in the single-phase ferrite temperature range.

(5) These alloys are susceptible to a variety of potential embrittlement mechanisms but, provided sufficient care is exercised, these should not present a problem in practice.

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