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The development of a tough high chronmium ferritic stainless steel

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Demo[44] investigated three techniques for controlling the interstitial content of high chromium (20Cr and 35Cr) stainless steels as a means of obtaining good as-welded properties. These techniques were

(i) keeping the interstitials (carbon and nitrogen) below critical levels,

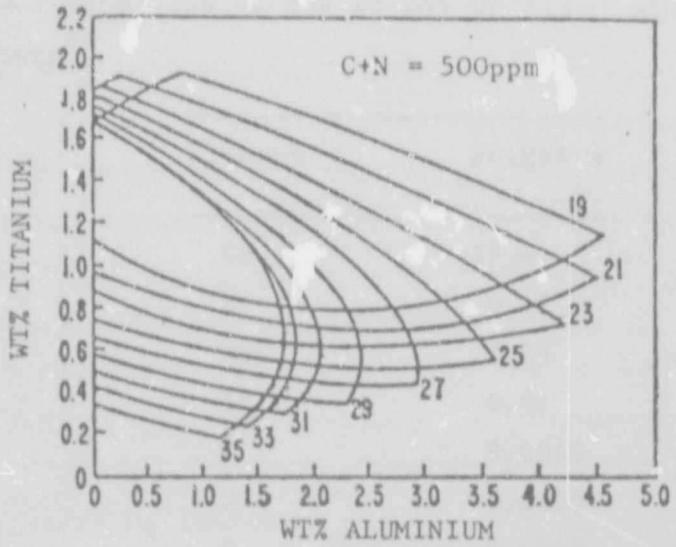
(ii) the addition of weld ductilisers, and

(iii) the addition of 'getterers' to complex the interstitials.

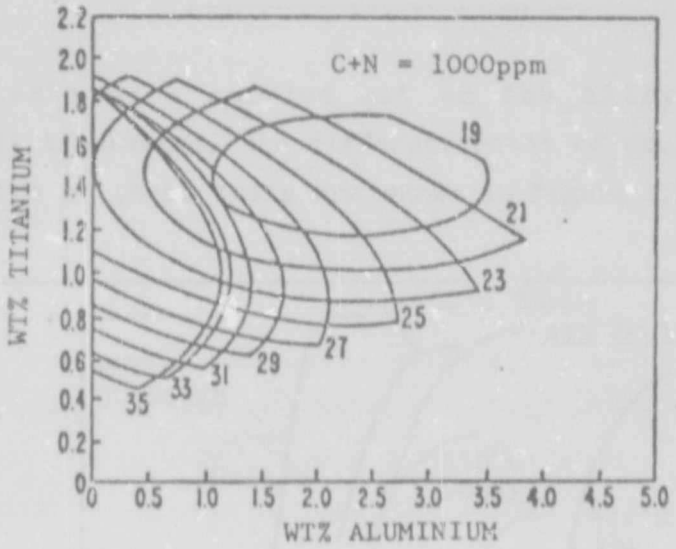
For good as-welded ductility, it was found that carbon and nitrogen levels of below 150ppm were needed in alloys containing more than 25% Cr. When 0,1 to 0,13% weld ductility additives (Al, Cu, V, Pt, Pd, and Ag) were used singly or in combination, ductile as-welded alloys could be produced, with chromium contents ranging from 28 to 37% and carbon and nitrogen contents up to 700ppm. By combining Al and Ti in a gettering action, high-chromium alloys were produced which had both as-welded ductility and corrosion resistance at higher levels of carbon and nitrogen than was possible previously. A statistically designed alloy programme was conducted and curves which defined 'good' areas of weld ductility and corrosion resistance as a function of composition were developed. These are shown in Figures 2.16 (a and b). The isochromium curves delineate alloy compositions with acceptable corrosion resistance and as-welded ductility at room temperature.

More recently a number of investigations have sought clarification of the role of carbon and nitrogen in brittle fracture. Carbon and nitrogen were studied in the dispersed solute, grain boundary-precipitated and intragranular-precipitated states.

Plumtree and Gullberg[45] showed that the impact transition temperature of an Fe-25Cr alloy in the annealed and water-quenched condition increased linearly with total dissolved interstitial content. The composition of their experimental alloy is shown in Table 2.4.

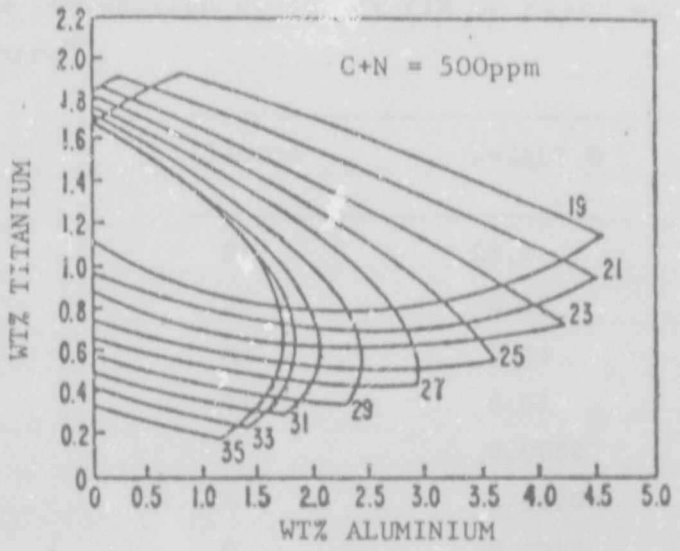


(a)



(b)

Figure 2.16 The effect of carbon and nitrogen gettering additives, Al and Ti[44].



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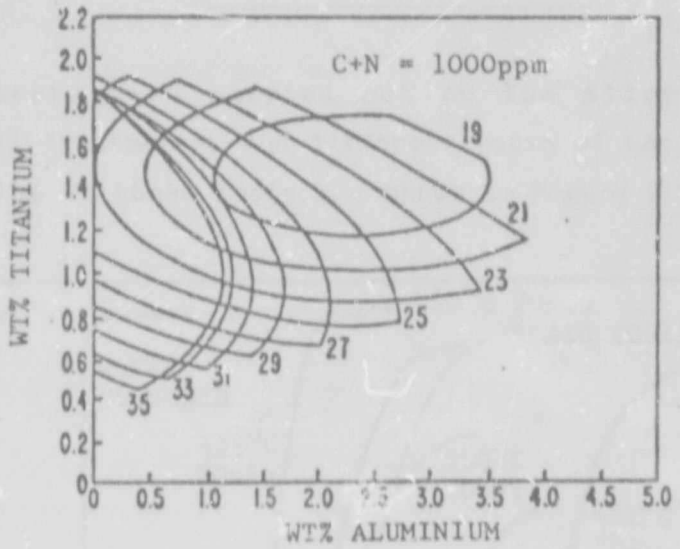


(b)

Figure 2.16 The effect of carbon and nitrogen gettering additives, Al and Ti[44].

Table 2.4 Composition of the Fe-25% Cr Alloy studied by Plumtree and Gullberg[44].

Element	Weight %
Cr	25,6
Ni	0,12
Mo	0,05
Ti	0,01
C	0,0030
N	0,0120
O	0,0200
Fe	balance

Impact tests were carried out on the alloy after various isothermal treatments, and different rates of cooling from 850°C. The results of these tests are shown in Figure 2.17.

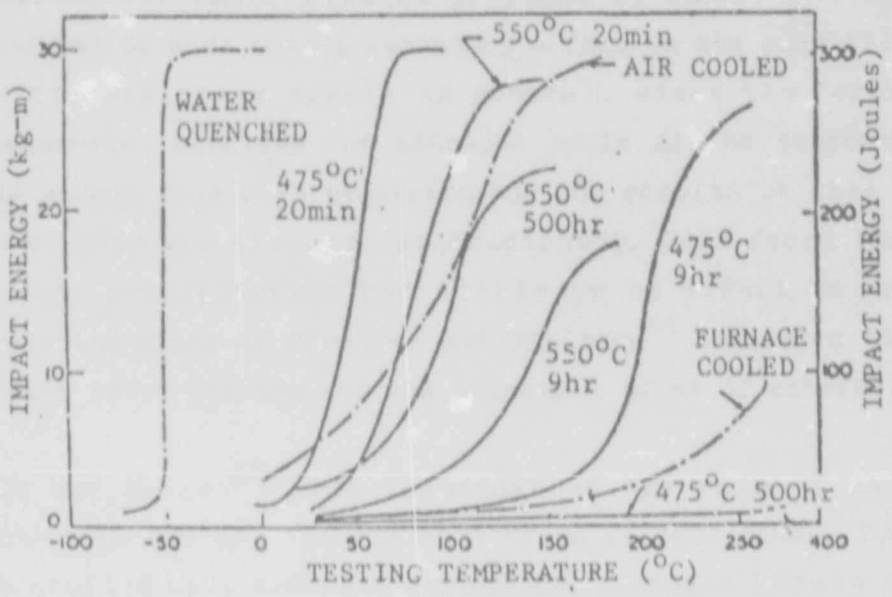


Figure 2.17 Impact energy curves for an Fe-25Cr alloy after various isothermal treatments (475 and 550°C) and different cooling rates from 850°C[45].

It was proposed that the embrittlement of their Fe-25Cr alloy (which had a relatively low interstitial content) was associated with the formation of grain boundary chromium nitrides and carbides. Slower cooling promoted grain boundary precipitation whereas more rapid cooling resulted in dispersed intragranular precipitation or retention of the carbon and nitrogen in solution. The effect of the interstitials in solution on the toughness of the alloy was benign in comparison with that of the carbide and nitride precipitates. The results were interpreted in terms of the Cottrell model for a ductile-to-brittle transition. The generally known fact that k_y increases with decreasing cooling rate[8,45] was quoted and this effect was attributed to the greater density of precipitates at the grain boundaries. The higher hardness of the more slowly cooled alloys was indicative of an increase in σ_y, although σ_o was expected to decrease as a result of the decrease in the solute atom drag. This effect was outweighed by the increases in k_y and σ_y and brittle fracture was favoured. Furthermore, it was suggested that the locked dislocation mechanism for embrittlement proposed by Demo[41,44], should be considered to have only a secondary effect on the embrittlement of ferritic stainless steels in general, since the formation of chromium-rich carbides and nitrides could not be suppressed by a rapid quench from high temperatures. The results of this work and that of Demo are clearly contradictory. Demo found that grain boundary precipitation had little or no effect on ductility whereas the study of Plumtree and Gullberg[45] indicated that grain boundary precipitation was the principle cause of embrittlement.

Grubb and Wright[20] also investigated the role of carbon and nitrogen in the brittle fracture of an Fe-26Cr steel. Two alloys were studied with combined carbon and nitrogen levels of 67 and 579 ppm respectively. Through heat treatment alone DBTT variations of greater than 200°C were achieved for each alloy. The heat treatments and results of the Charpy tests are shown in Tables 2.5 and 2.6, respectively.

At the 67 ppm level, rapid quenching suppressed carbide and

Table 2.5 Heat Treatments used to establish varied states of C and N^{20}.

Procedure	Description
A	1290°C for 1 min;spray quench
B	1290°C for 1 min;iced-brine quench
C	1290°C for 1 min;air cool
D	1290°C for 1 min;furnace cool
E	1290°C for 1 min;iced-brine quench; and 955°C for 10 min;iced-brine quench
F	1290°C for 1 min;iced-brine quench; and 870°C for 10 min;iced-brine quench
G	1290°C for 1 min;iced-brine quench; and 790°C for 10 min;iced-brine quench
H	1290°C for 1 min;iced-brine quench; and 705°C for 10 min;iced-brine quench
I	1290°C for 1 min;iced-brine quench; and 620°C for 10 min;iced-brine quench
J	1290°C for 1 min;iced-brine quench; and 540°C for 10 min;iced-brine quench

nitride precipitation completely and a DBTT of -130°C was achieved (A and B). Carbide and nitride precipitation was then induced (through isothermal annealing at low and intermediate temperatures or through slow cooling) and the DBTT became considerably higher (C,F,G,H,I and J). Carbide precipitation was observed to occur above 850°C whereas nitride precipitation developed at much lower temperatures (600°C and lower). In this alloy the cause of embrittlement was identified as grain boundary precipitation, which produced an increase of 50 to 70°C in the DBTT (F,G and H). A DBTT value as high as 90°C (procedure D) was associated with intergranular plate-like precipitation of Cr_2N, and the procedure E which resulted in fine intergranular stippling but no grain boundary precipitation, produced the lowest DBTT.

Table 2.6 Charpy V-notch transition temperatures and upper shelf energies, 0,155cm thick specimens[20].

	Heat I 67ppm (C and N)		Heat II 570ppm (C and N)	
Procedure	DBTT, C	Upper Shelf Energy,J	DBTT, C	Upper Shelf Energy,J
A	-130	16	75	14
B	-130	20	100	15
C	-100	18	80	14
D	90	C	205	9
E	-135	17	115	10
F	-60	17	70	12
G	-85	18	50	18
H	-80	16	0	17
I	-115	20	115	16
J	0	18	100	14

For the 570 ppm (C + N) alloy, quenching to suppress carbide and nitride precipitation (A and B) resulted in severe embrittlement (DBTT 100°C). Grain boundary and plate-like precipitates still appeared to be the main sources of embrittlement (procedures C to J). The lowest DBTT of 0°C (H) was associated with a mixture of grain boundary and intragranular precipitates and the highest, 205°C (D), with heavy grain boundary precipitation produced by furnace cooling.

It was observed that the Cr_2N precipitates opened readily during plastic deformation. It was suggested that these precipitates promoted brittle fracture by lowering the effective surface energy of the crack, γ. Furthermore, they increased σ_o and hence σ_y. It should be noted that the role of the plate-like precipitates in fracture was significant only for the 67ppm (C+N) alloy. In the 570ppm (C+N) alloy, microcracking was associated with twin-grain

boundary impingement, cracking at the grain boundary film-matrix interface and cracking of the grain boundary precipitate itself. No direct TEM evidence for dislocation immobilisation by precipitates was found for either material.

More recent studies[1,2,32,38,45,46,47] have not shed any new light on the role of interstitials in brittle fracture.

Onashi et al[38] working on molybdenum-containing 18Cr, 26Cr and 29Cr stainless steels confirmed the results of previous investigators that steels with low interstitial levels exhibit good toughness in the solution heat-treated condition and are embrittled by precipitation of secondary phases when cooled or reheated at intermediate temperatures. The solution heat treatment temperatures used in their study ranged from 1000°C to 1200°C and the intermediate temperatures were in the range 700°C to 900°C.

Grubb[2] investigated the brittle fracture of a 26Cr stainless steel and also showed that secondary phases are detrimental to toughness properties and that quenched alloys with intermediate and high interstitial levels are severely embrittled by the precipitation of plate-like nitrides. The nitrides were observed to be very efficient initiators of microcracks. These results are similar to those obtained earlier by Grubb and Wright.

In 1984, Rodriguez[1] conducted fracture initiation studies on Fe-29Cr-4Mo-2Ni and Fe-26Cr-3Mo-2Ni stainless steels aged at 540°C. Neither of these alloys contained plate-like precipitates and fracture initiation was found to be related to mechanical twinning and titanium carbonitride precipitation at low temperatures.

The reasons why grain boundary precipitation is associated with poor ductility in some cases[2,20,38,48] and does not appear to affect the ductility in others[41] still remains to be elucidated.

2.1.5.2 Austenite and martensite

When carbon and nitrogen are inhomogeneously distributed in an alloy, small patches of austenite may occur in areas of relatively high concentration. The austenite is either retained, or transformed to martensite on cooling.

Austenite and martensite contribute to the embrittlement of ferritic stainless steels. Semchyshen et al[42] found that 17% chromium alloys, with carbon and nitrogen contents of 0,061% and 0,057% respectively contained a considerable amount of untempered martensite on quenching from 1150 C. They considered this martensite to be at least partly responsible for the high DBTT (>100 °C) of the alloys. This can occur by the hard martensite plates concentrating stress in the relatively soft ferrite matrix[8]. Microcracks are more easily initiated or propagated in these highly stressed areas, and the fracture process then requires less energy. Demo[41] considered embrittlement at high temperatures to be related in part to retention of austenite itself which may also cause large localised stress concentrations in the matrix. However, little work has been done to quantify the effects of austenite and martensite on the DBTT.

2.1.6 Other factors which influence the DBTT

2.1.6.1 Stress state

Saito et al[47] showed that the fracture behaviour of a high purity Fe-30Cr-2Mo alloy was strongly dependent on the conditions of hydrostatic tensile stress. Under conditions of low triaxial stress the alloy displayed excellent ductility while for high triaxial stresses greater than 80kg/mm^2 (785MPa) it showed less strain to fracture and a transition from ductile to cleavage fracture. This type of behaviour was found to be more pronounced in high chromium alloys than in plain carbon steels[47].

2.1.6.2 **Grain size**

Saito et al[47] also showed that the toughness of the Fe-30Cr-2Mo alloy can be significantly improved by thermomechanical processing. The main effect of the processing was to alter the grain size. Table 2.7 lists the processing conditions, and the corresponding grain sizes and impact energies.

Table 2.7 Effect of processing condition on ferrite grain size

Processing	Temperature (°C)			ASTM Grain size	Hardness, Hv	Impact energy	
	Finishing	1st Anneal	2nd Anneal			kgm/cm^2	(J/cm^2)
A	1000	-	1000	1 to 2	208	-	-
B1	700	-	1000	3 to 7	203	10	(98)
B2	700	850	1000	3 to 7	210	5	(49)
C1	620	850	-	8 to 9	208	28	(275)
C2	620	850	1000	7 to 6	212	-	-
D	400	720	1000	8	208	32	(314)
E	20	720	1000	8	232	30	(294)

Comparison of the data in the table shows that finer grain sizes were promoted by low finishing temperatures (on the final rolling pass) and a suitable first annealing temperature (to activate nuclei for recrystallisation prior to the final anneal). Annealing times were very short: 10 or 20 minutes for the first anneal and only 5 minutes at 1000°C. The finer grain sizes were associated with larger impact energies.

Other investigators[4,28,38,46] found that grain size variations had little effect on toughness properties. Mentz[46] provided an explanation for the discrepancy. It was suggested that the main source of variability was related to the difficulty in isolating

the influence of grain size from other variables which affect impact behaviour, such as carbide thickness, precipitation and solute hardening, and the presence of fissure-like flaws.

2.1.6.3 Cold work

The way in which cold work affects the kinetics of σ-phase formation was discussed in Section 2.1.4.1. In addition to accelerating the formation of σ, Vigor[49] found that cold work had ...d effect on the DBTT of a 409 stainless steel (which has ...position: 11% Cr, a titanium stabiliser and the balance ...old reduction of 15% raised the DBTT of the stee... 40°C. However, the effect of cold work on the DBTT was fou... be related to the carbon content of the alloy. AISI 409 contains a maximum of 0,08% C and the DBTT of a similar alloy containing only 0,03% C was shown to be less sensitive to cold work.

2.1.6.4 Notch Sensitivity

Annealed iron-chromium stainless steels are highly notch sensitive[50]. As early as 1935, Krivobok[50] showed that the room temperature impact resistance of these alloys declined sharply in the presence of a notch when the chromium content exceeded 15%. This decline was independent of carbon content in the range 0,01 to 0,02%.

In 1980, Onashi et al[38] published the results of work on Fe-26Cr-Mo alloys in which the effect of notch sharpness on the DBTT was investigated. Three types of notches (a 2mm V-notch, a fatigue crack and a brittle cold crack) were made in full-size Charpy specimens. The results of the impact tests are shown in Figure 2.18.

As the sharpness of the notches increased so the DBTT increased. The difference in DBTTs for the embrittled and annealed specimens diminished as the notch size decreased. These results suggest that solution-treated steels are tougher than the steels containing

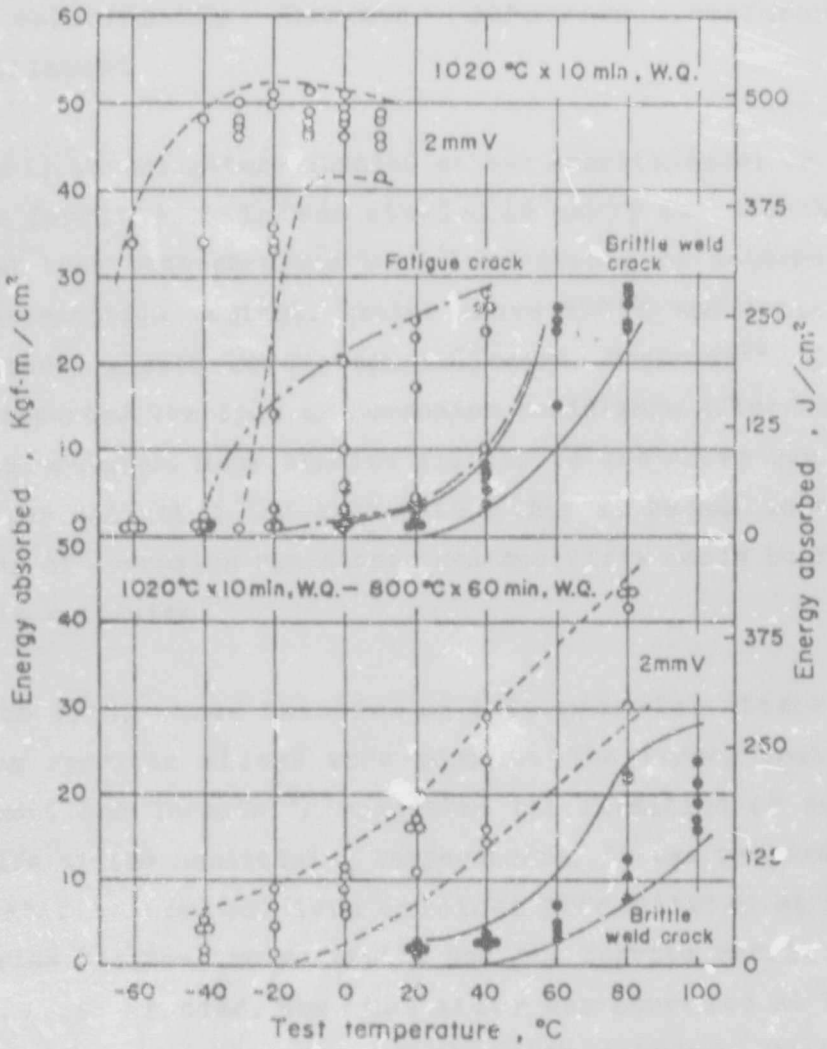


Figure 2.18 Effects of notch sharpness on Charpy impact toughness of 26Cr-1Mo steel specimens solution treated at 1020°C or embrittled at 800°C for 1 hour. Open circles - V-notched samples, semi-solid circles - fatigue cracked samples and solid circles - brittle weld cracked alloys[38].

precipitates because they have better resistance to brittle crack initiation viz. the more blunt the notch in a solution-treated steel, the more difficult it is to initiate a crack. In contrast the precipitates in the other steels act as crack initiators. The resistance to crack propagation is, however, similar for the steels in either condition as indicated by the results for the brittle cold cracked specimens.

2.1.7 The relationship between corrosion resistance and embrittlement

Most early investigators studied either embrittlement or corrosion loss in ferritic stainless steels. As early as 1951 Thielsch[51] reported that high-chromium stainless steels of intermediate and high interstitial content, heated above 950°C and cooled to room temperature, showed severe embrittlement. Hochmann[52], in another study, reported the loss of corrosion resistance after exposure to high temperatures. Many studies similar to the above can be cited, yet it was not until the late 1960's that it became accepted that the loss of corrosion resistance and ductility could be related to a single mechanism.

Prior to 1960, three theories on intergranular attack in high-chromium ferritic alloys were popular. The first, postulated by Houdremont and Tofaute[53], suggested the formation of carbon-rich austenite at the sensitising temperatures. It was proposed that as the austenite cooled, iron carbides precipitated at the grain boundaries between the austenite and the ferrite matrix and these were easily corroded. When the alloy was annealed at 750°C the iron carbides transformed to chromium carbides which resist chemical dissolution and the material became resistant to intergranular corrosion.

The second, a proposal by Hochmann[52], also involved austenite formation at high temperatures but suggested that intergranular attack occurred preferentially at the boundaries of the austenite grains themselves because of their low chromium and high carbon contents.

The third theory, which was formulated by Lula, Lena and Kiefer[54], rejected any mechanism of intergranular corrosion that required the formation of austenite. It was pointed out that operations aimed at preventing austenite formation during high temperature exposure did not prevent intergranular corrosion. These investigators proposed that the stress surrounding carbides or

nitrides which precipitated during cooling, sensitised the matrix.

By the early 1960's it was recognised that high temperature embrittlement and loss of corrosion resistance were related phenomena. In 1963, Baumel[55] suggested that loss of corrosion resistance at high temperatures was due to chromium depletion adjacent to the chromium carbide precipitates which also embrittled the alloys.

In the decade which followed a number of investigators[4] confirmed that the chromium depletion theory was the most plausible explanation for severe intergranular attack. Demo[56] reported the effects of heat treatment on the corrosion resistance of a commercial AISI type 446 stainless steel. Specimens of this material showed poor corrosion resistance when quenched or air-cooled from 1100°C. However, these sensitised specimens recovered when reheated to 850°C. Other specimens which were slow cooled in the furnace from 1100°C to temperatures below 1000°C and then quenched, had a resistance to corrosion equivalent to that of the annealed specimens.

Bond[57] described the effects of carbon and nitrogen levels on the sensitisation of a series of 17% chromium alloys. Some of Bond's results are shown in Table 2.8.

It can be seen from this table, that temperatures of 925°C and higher caused sensitisation in alloys which contained more than 0,022% N or 0,012% C. Furthermore, sensitisation occurred at temperatures which correspond to a marked increase in the solubility of carbon and nitrogen in ferrite. On cooling, the ferrite thus became supersaturated with carbon and nitrogen and chromium-rich carbides or nitrides precipitated.

Bond concluded that this sensitisation was best explained by the chromium depletion theory. Furthermore, it was noted that the precipitation reactions which occurred at these high temperatures were so rapid that even quenching seldom suppressed them[57].

Table 2.8 Results of the boiling 65% nitric acid test on selected 17Cr alloys containing carbon and nitrogen[57].

Carbon,%	Nitrogen,%	Corrosion Rate, mm/dm^2/day, for the average of 5 successive 48hr periods after 1hr of heat treatment at the indicated temperature, water quench			
		788°C	926°C	1038°C	1150°C
0,0021	0.0095	167	185	148	326
0,0025	0.0220	164	944	-	761
0,0044	0,0570	186	-	577	357
0,0120	0,0089	126	824	1817	-
0,0610	0,0071	147	619	1574	-

Following work on the effects of isothermal heat treatment on a high purity Fe-26Cr stainless steel, Hodges[58] proposed that the loss of corrosion resistance at 475°C was also related to chromium depletion adjacent to grain boundaries. It was also shown that molybdenum additions could delay the onset of this sensitisation.

In a continuation of his earlier work, Demo[41] published the results of further studies on commercial high chromium AISI steels, types 446 (27%Cr) and 304 (18%Cr), and an experimental alloy (26%Cr), the compositions of which were given in Table 2.2.

The reasons for the loss of corrosion resistance and ductility following exposure to high temperature were investigated and a single mechanism for both phenomena was proposed.

The results of the corrosion tests and the micrographs of the samples are shown in Figure 2.19.

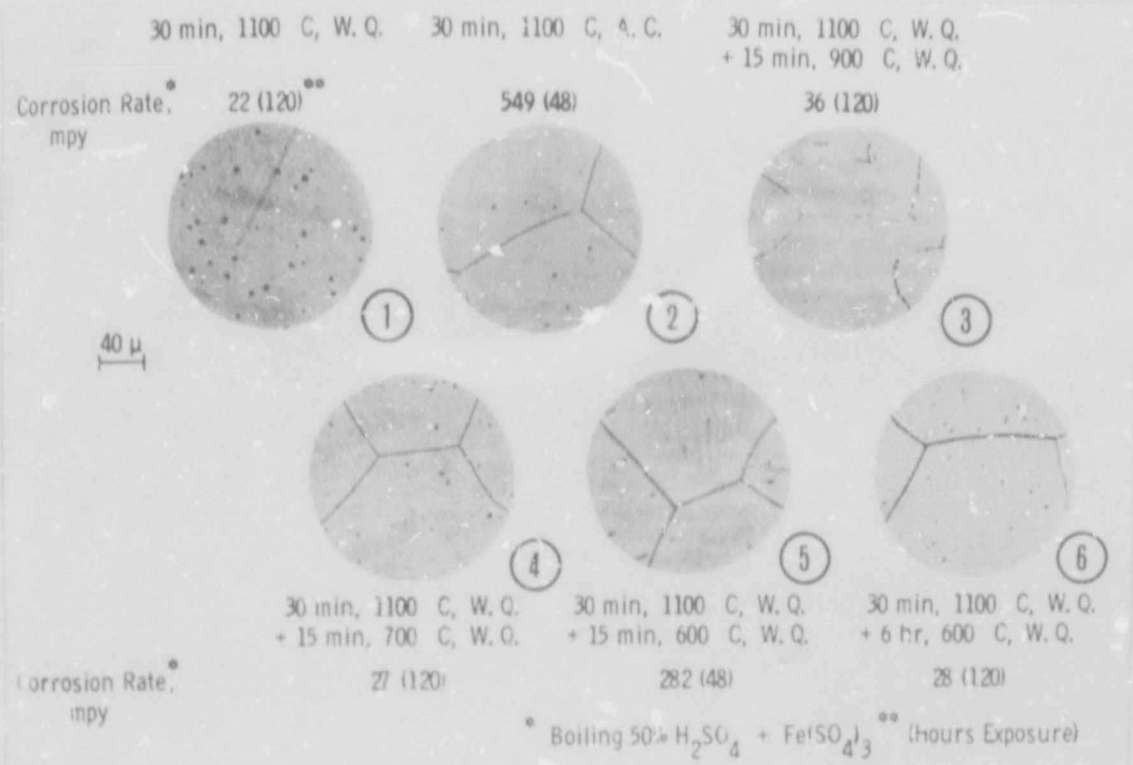


Figure 2.19 Effect of thermal treatment on the microstructure and corrosion resistance of experimental Fe-26%Cr alloys (C+N = 180ppm)[41].

From the corrosion and microstructural data Demo constructed a time-temperature sensitisation envelope (TTS) as shown in Figure 2.20.

The diagram is explained as follows. When ferritic stainless steels are heated above 950°C, chromium-rich carbide and nitride precipitates are dissolved. If the interstitial levels are very low, the interstitials are maintained in solid solution on quenching and good corrosion resistance and ductility are obtained. In the temperature range 700 to 950°C, chromium diffuses into the chromium-depleted areas as soon as precipitation occurs. Consequently, the corrosion resistance of the alloys remains good but the ductility of the alloys suffers as a result of precipitation at the grain boundaries and on dislocations (see earlier discussions, Section 2.1.5.1.). In the temperature range 500 to 700°C in which chromium-rich carbides and nitrides

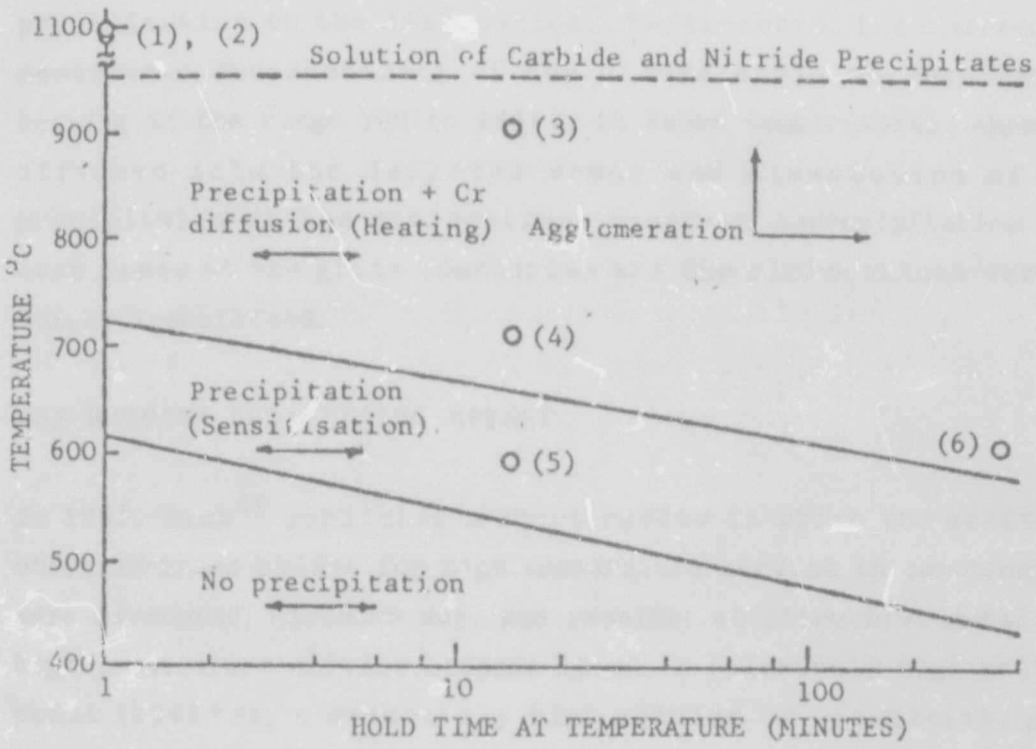


Figure 2.20 TTS diagram for Fe-26Cr alloys treated initially at 1100°C, 30 min, water quenched, then reheated at selected lower temperatures for different times: corrosion tested in boiling ferric sulphate - 50 percent sulphuric acid solution: (C+N = 180ppm)[41].

precipitate rapidly, the chromium-depleted zones are not healed except during long hold times because the diffusion rate of chromium is low. Both corrosion and ductility are poor after heat treatment at temperatures in this range.

For high chromium alloys of intermediate and high interstitial levels, rapid quenching from temperatures above 950°C is not fast enough to prevent precipitation of chromium-rich carbides and nitrides. Thus chromium-depleted areas develop because the alloys do not dwell long enough at temperatures which are sufficiently high for chromium diffusion. As a result, the corrosion resistance as well as the ductility of these quenched alloys are poor.

Demo[41] found that the loss of ductility in the alloys with

intermediate and high interstitial contents, was associated with precipitation on the dislocations. Furthermore, both corrosion resistance and ductility of the alloys could be restored by heating in the range 700 to 950°C. At these temperatures, chromium diffused into the depleted areas and dissolution of the precipitates at the dislocations occurred. Reprecipitation then took place at the grain boundaries and the dislocations were no longer immobilised.

2.2 THE RHENIUM DUCTILISING EFFECT

In 1963, Sims[59] published a short review in which the merits of chromium-based alloys for high temperature service in construction were discussed. Chromium was, and remains, an attractive metal for high temperature service because it has a relatively high melting point (1849°C), a moderately high modulus of elasticity, good thermal shock resistance and superior oxidation resistance[44,40]. The major disadvantage of chromium and high-chromium alloys is their sensitivity to interstitial elements such as nitrogen which, when present even in small amounts, drastically reduce ductility. For example, very pure chromium (99,99%) has a UTS of 415 MPa and an elongation of 44%, whereas chromium which is 99,95% pure has a UTS of up to 689 MPa depending on processing and a maximum elongation of only 10%[59,60]. Nevertheless Sims predicted that the availability and inexpensiveness of chromium would give impetus to the advancement of chromium-related technology and that chromium-based alloys of useable ductility, strength and superior oxidation resistance would eventually become available commercially.

By 1968, it was already well established that the DBTT of group VI-A metals (Cr, Mo, and W) could be substantially reduced by alloying with 25 to 35% Re[61]. This phenomenon is referred to as the rhenium ductilising effect. It was first observed in 1955 by Geach and Hughes[62] who found that alloys of W-25Re and Mo-35Re possessed considerably improved fabricability and low temperature ductility compared with unalloyed tungsten and molybdenum. Klopp[63], in a later study, showed that Cr-35Re also exhibited

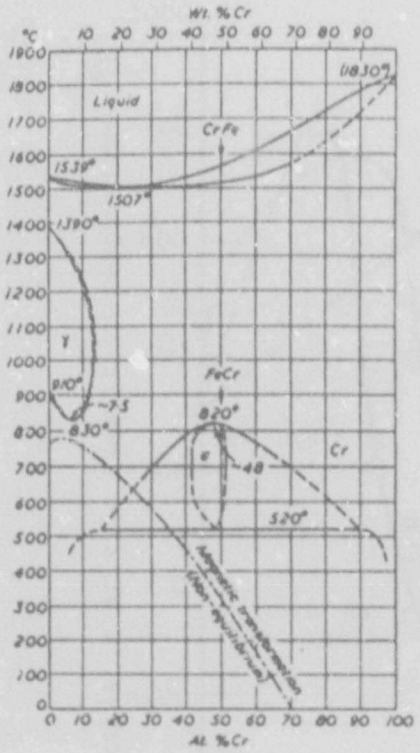
improved fabricability and ductility in a similar manner.

The generality of this effect was explored by Stephens and Klopp[61] in their work directed at improving the ductility of chromium through alloying. Various chromium alloys were studied using alloying additions (such as Ru, Co, Mo, Mn and Fe) which had phase diagrams with chromium similar to the Re-Cr diagram. All the phase diagrams show high solubility of the solute in chromium and an intermediate σ-phase. Some of the phase diagrams are shown in Figure 2.21.

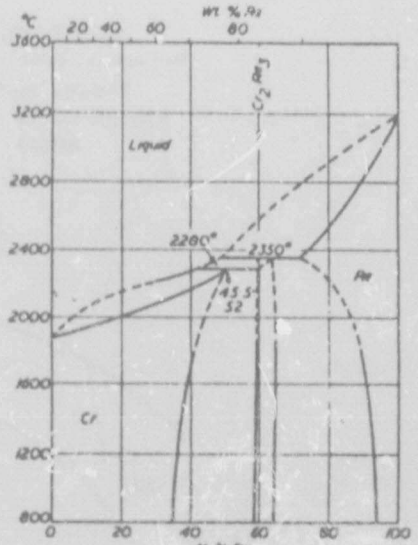
Seven other systems: Cr-Ti, Cr-V, Cr-Nb, Cr-Ta, Cr-Ni, Cr-Os and Cr-Ir which partly resemble Cr-Re were also studied[61]. They have either extensive solid solutions or form complex intermediate phases which are not necessarily σ. These alloys (which do not have phase relations with extensive solid solution and an intermediate σ-phase) were found to be unfabricable or brittle. Their DBTTs were higher than that of unalloyed chromium.

Alloys with lower DBTTs than unalloyed Cr (149°C) included Cr-35Re, Cr-40Ru, Cr-27Ru, Cr-30Co, Cr-40Fe and Cr-50Fe. The results of the 3-point bend tests for the Cr-Re, Cr-Ru, Cr-Co and Cr-Fe alloys are summarised in Figure 2.22.

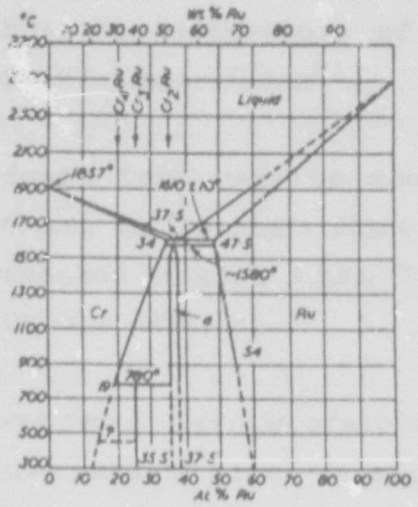
It was noted that when the compositions approached the solubility limit in the Cr-Re, Cr-Ru and Cr-Co systems, and the sigma composition in the Cr-Fe system, the DBTT decreased significantly. The alloys with compositions closest to maximum solubility had the lowest DBTTs.



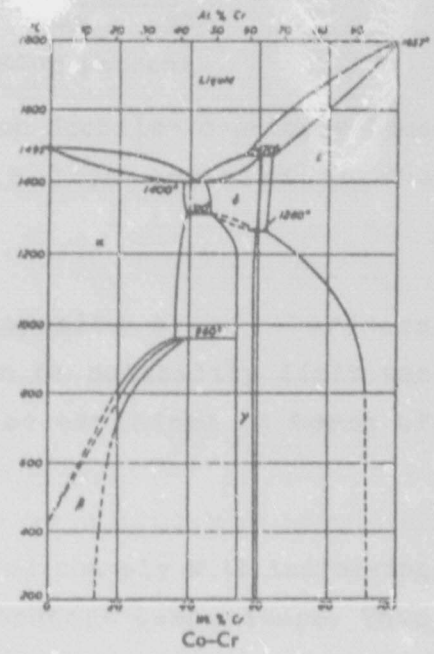
Cr-Fe



Cr-Re



Cr-Ru



Co-Cr

Figure 2.21 Equilibrium phase diagrams for binary chromium alloy systems[60,51].

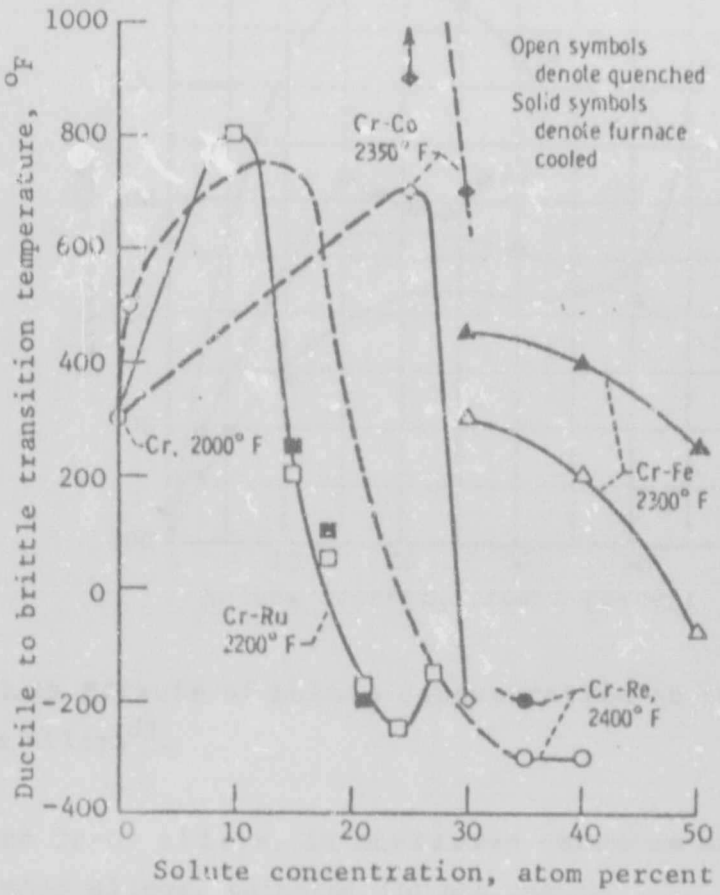


Figure 2.22 Effects of solute content on ductile-to-brittle bend transition temperature of chromium alloys annealed at the indicated temperatures[61].

Hardness test results showed the opposite trend: hardness decreased as the sigma phase composition or solubility limit was approached (Figure 2.23). This could be explained in terms of grain size effects.

It was found that the grain size increased sharply with increasing solute content after annealing at a constant temperature. This effect is shown in Figure 2.24.

Metallographic investigation revealed that the initial mode of deformation primarily involved twinning. In the Cr-Fe alloys, as the iron content increased from 30 to 50% the number of twins decreased and the width of the twins increased. Both these effects were also observed as the solute content increased in the Cr-Co,

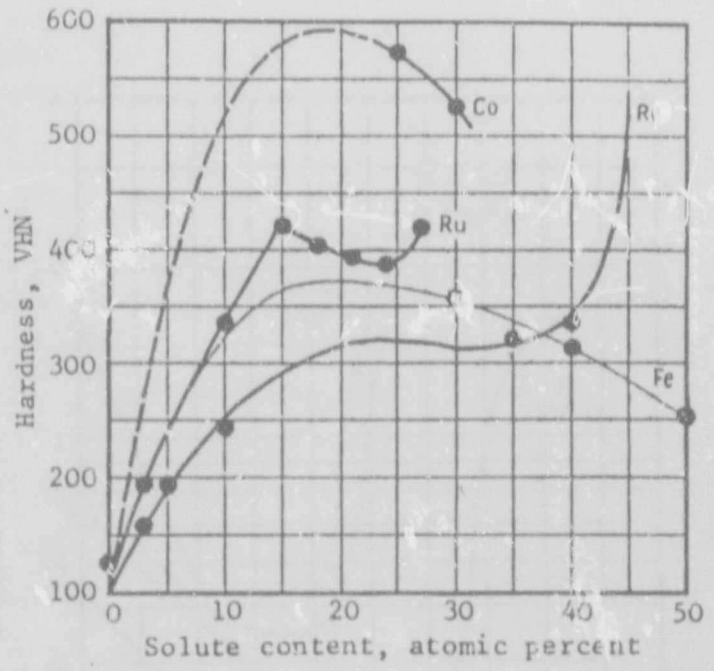


Figure 2.23 Effects of solute concentration on the hardness of chromium alloys[61].

Cr-Ru and Cr-Os alloys. In unalloyed chromium and in the less concentrated alloys, twinning did not occur.

Klopp and Stephens concluded that phase relations were important in alloys which display the rhenium ductilising effect. They suggested that a high solubility, an intermediate σ-phase and a composition near the maximum solubility contributed to the decrease in the DBTT of chromium by favouring a metastable structure with respect to σ precipitation. During quenching or straining at lower temperatures, this metastable structure produces localised stresses and acts as a source for dislocations and twins. Deformation is then easier and the DBTT is lowered as a result. The increase in grain boundary mobility and exaggerated grain growth at solute contents near to the maximum were cited as evidence of the metastability of the structure.

Although Klopp and Stephens were primarily concerned with ductilising chromium metal, their results also confirmed the observations of other investigators working in the field of ferritic stainless steel development (see Section 2.1.5). For

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