PROCESSING AND PROPERTIES OF INTERSTITIAL-FREE STEELS

G. Krauss, D.O. Wilshynsky, and D.K. Matlock

Advanced Steel Processing and Products Research Centre
Colorado School of Mines
Golden, CO, USA 80401

Abstract - This paper reviews the physical metallurgy of vacuum-degassed, interstitial free, cold rolled and annealed sheet steels. The effects of chemical composition and processing on formability are tracked by changes in the normal plastic anisotropy factor, r_m. The evolution of precipitate dispersions and ferrite are related to differences in the contribution of the Nb and Ti additions used to remove interstitial carbon from solid solution and to control the development of favorable ferrite texture during recrystallization. The different effects of Nb and Ti evolve throughout the entire thermomechanical processing schedule, including slab reheating, hot rolling, coiling, cold work and annealing. The effects of Mn, S, and P are also discussed selective to solid solution strengthening, cold work embrittlement, and aging.

INTRODUCTION

The production of steels with very low C and N contents constitutes the most recent step in the evolution of formable, cold-rolled and annealed steels. Low carbon content and additions of Al, Nb, and Ti which combine with interstitial elements have long been known to promote recrystallization textures favorable for severe forming operations such as deep drawing (1,2). However, steelmaking in oxygen converters, although capable of producing N contents on the order of 20 ppm (0.002 wt pct), typically can reduce C only to 100 to 200 ppm (0.01 wt pct-0.2 wt pct) (3). Further decreases in C contents, to values as low as 30 to 50 ppm require vacuum degassing and rigorous control of C, N, and O pick-up during casting. Such state-of-the-art processing steps are now commonly applied and have led to the availability of a new class of steels, variously referred to as interstitial-free (IF) steels, vacuum-degassed IF steels, ultra-low carbon (ULC) steels or extra-low carbon (ELC) steels (4-6).

Table 1 shows typical composition ranges of the elements in vacuum-degassed interstitial-free steels, and Figure 1 shows the Fe-rich side of the Fe-C diagram (7). The low carbon contents of IF steels are well below those of steels which form pearlite after hot rolling or distributions of spheroidized carbide particles after cold rolling and annealing. Despite the low interstitial levels of ULC steels, the residual carbon may affect texture formation, and the low solid solubility of C in bcc α-iron, as shown in Figure 1, may cause strain or quench aging in annealed microstructures. For these reasons, Nb and Ti are added to IF steels to completely remove C and N from interstitial solid solution by forming stable compounds of Nb and Ti. Figure 1 also shows that the A_1 and A_3 temperatures of IF steels are higher than those of higher carbon steels. This increased range of ferrite stability affects temperatures used for finish hot rolling and for annealing after cold rolling.

TABLE 1 - Composition (wt pct) IF DDQ Steels (from 4,5,6)

C	Si	Mn	P	Al	N	Nb	Ti	S
0.002- 0.008	0.01- 0.03	0.10- 0.34	0.01- 0.02	0.03- 0.07	0.001- 0.005	0.005- 0.040	0.01- 0.11	0.004 0.01

The key to the enhanced formability of IF steels is the formation of favorable {111} <1$\bar{1}$0> (cube-on-corner) recrystallization textures (8,9). Microstructures with these textures have high values of the plastic strain ratio, r, defined as follows (1):

$$r = \epsilon_w/\epsilon_t = \ln(w_i/w_t)/\ln(t_i/t_t) \qquad (1)$$

The r values are a function of orientation, and the anisotropy of r values is incorporated into average terms, r or r_m, as follows:

$$\bar{r} = r_m = (r_o + 2r_{45} + r_{90})/4 \qquad (2)$$

where r_o = r value determined in the rolling direction, r_{45} = r value determined at 45° to the rolling direction, and r_{90} = r value determined in the transverse direction.

Another measure of formability, related to earring, is the planar anisotropy defined as follows:

$$\Delta r = (r_o - 2r_{45o} + r_{90o})/2 \qquad (3)$$

Microstructures with high r_m values are desirable because they resist the thinning which results in fracture of sheet subjected to severe forming deformation. The r_m values of stabilized IF steels are often above 2.0, a significant improvement relative to higher carbon sheet steels. Thus, although vacuum-degassed IF steels also have low flow stresses and high tensile ductility, both factors which promote good formability, the r_m values are almost universally used to evaluate the performance of IF steels.

In addition to the stabilizing elements Nb and Ti, all of the elements listed in Table 1, depending on their microstructural interactions in the various thermomechanical processing steps, may significantly affect the optimization of texture, microstructure and properties of cold-rolled and annealed IF steels. The following sections briefly describe some specific effects of chemistry and processing. The effects of chemistry and processing, however, are deeply interwoven and the effects of one parameter often cannot be isolated from interactions with other elements or processing steps.

ALLOYING FACTORS

Carbon and Nitrogen

The driving force for producing IF steels is the beneficial effect which very low C and N contents have on formability. Figure 2 shows r_m as a function of carbon content for a number of steels, as compiled by Hutchinson et al. (10). Figure 3, after early work by Fukuda (11), couples the effect of C and cold rolling deformation on the development of r_m values. Both curves demonstrate the very positive effect of low C on r_m. The high r_m values are associated with the formation of beneficial recrystallization textures which are produced after hot deformation, coiling, cold rolling and annealing. A critical fundamental observation in this processing sequence is the fact that carbon atoms in interstitial solid solution degrade beneficial {111} recrystallization texture and increase unfavorable {110} and {100} components (9). In higher carbon steels with coarse cementite particles, ferrite with undesirable orientations may nucleate on the particles, but the mechanism by which interstitial carbon affects recrystallization is not fully understood (9).

Nitrogen is also a critical interstitial element, but at the low levels present in IF steels is considered to be effectively removed from interstitial solid solution by aluminum nitride or titanium nitride formation. As a result, major attention is focused on the removal of carbon by steelmaking, as noted earlier, and by stabilizing alloy additions, as discussed below.

Titanium and Niobium

Stabilizing additions of Ti and Nb affect the recrystallization process in two ways. Most important, these elements form carbide particles which remove C from interstitial solid solution. Also, the carbide and other compounds of Ti and Nb form particle distributions which restrain large angle grain boundary migration and therefore reduce the nucleation and growth rates of recrystallized grains (12). Therefore, in stabilized IF steels with low interstitial carbon content, preferentially nucleated {111} grains nucleate and grow, albeit at low rates, but the nucleation and growth of competing grains with other orientations is hindered even more (9).

In contrast to Al-killed steels, in which Al and N are kept in solution (by a combination of high slab reheat temperatures, high finishing temperatures, and low coiling temperatures), until batch annealing (1), Nb and Ti IF steels are alloyed and processed to form stable carbides during hot deformation processing prior to cold working. Table 2 lists a number of equations which have been developed for the temperature-dependence of the solubility products for Nb, Ti, and Al nitride, carbide, and carbonitride formation in austenite (13,14). There is considerable variation and overlap between the expressions for the various compounds, but the table consistently shows a very strong temperature dependence for TiN formation. Thus the solubility of Ti as related to TiN formation is expected to drop very rapidly with temperature, resulting in TiN precipitation, and less Ti available for TiC formation. Ti also combines with sulfur, in the form of $Ti_4S_2C_2$. Thus the amounts of Ti required to stabilize IF steels are given by the following equations (15):

$$Ti_{eff} = \text{Total Ti} - 3.42N - 1.5S \quad (1)$$
$$Ti^* = \text{Total Ti} - 4C - 3.42N - 1.5S \quad (2)$$

where Ti_{eff} is the in solution after combination with N and S,and Ti* is the Ti remaining in solution after combination with N, S, and C. Figure 4 shows that Ti above the amount required to combine with all N, S, and C is necessary to achieve high r_m values (16).

Niobium, when added to IF steels, is assumed to combine only with carbon, and therefore no corrections are made for the effects of other elements. Figure 5 shows the effect of Nb on the properties of a Nb-containing IF steel after cold rolling and annealing at 830°C. As in the case of Ti, Nb over the amount required to completely combine with carbon is necessary to achieve the highest r_m values. The aging index (AI), a mechanical measure of the amount of interstitial carbon available for strain aging after hot rolling, coiling, cold working and annealing, is zero, indicating that carbon, even at Nb/C ratios less than one, is no longer solid interstitial in solution after processing.

The temperature dependence of solution (on heating) and the precipitation (on cooling) of the various compounds of Ti and Nb strongly affect the processing of IF steels. Generally the compounds of Ti dissolve and precipitate at higher temperatures than do Nb compounds. As a result the Ti precipitate dispersions are more stable, and play less of a role in the lower temperature portions (finish hot rolling, coiling, and annealing) of the processing schedules. This fundamental difference may contribute to the observation that Ti-stabilized IF steels are less sensitive to alloying and processing variations (5). The high stability of Ti compounds relative to NbC is demonstrated in Figure 6 which compares the effects of reheating on the dissolution of precipitates in Nb NL and Ti IF steels (17). Even at a slab-reheating-temperature (SRT) of 1000°C, all of the NbC is dissolved, while at 1250°C substantial amounts of Ti precipitates are still undissolved.

Manganese and Sulfur

The combination of Ti with sulfur and carbon has already been mentioned. Manganese and sulfur also combine in low carbon steels, and with an excess of Mn above that required to combine with the available S, r_m values decrease, as shown in Figure 7 (18). The latter effect is attributed to the impeded growth of {111} ferrite grain orientations by a Mn solute drag mechanism (10). There are apparently other factors

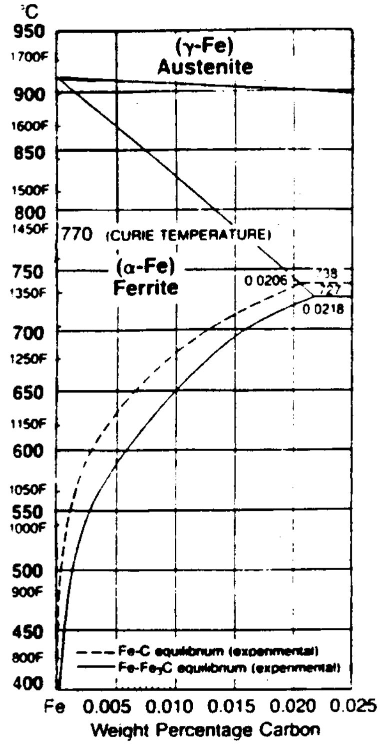


Fig. 1 - Fe-rich site of Fe-C diagram, showing extent of ferrite phase field and decrease of carbon solubility with decreasing temperature (from ref. 7).

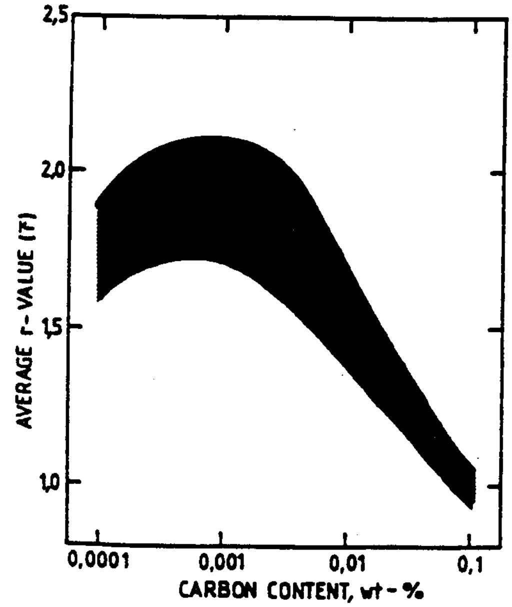


Fig. 2 - Effect of carbon content on r_m values in steel (from Hutchinson et al., ref. 10).

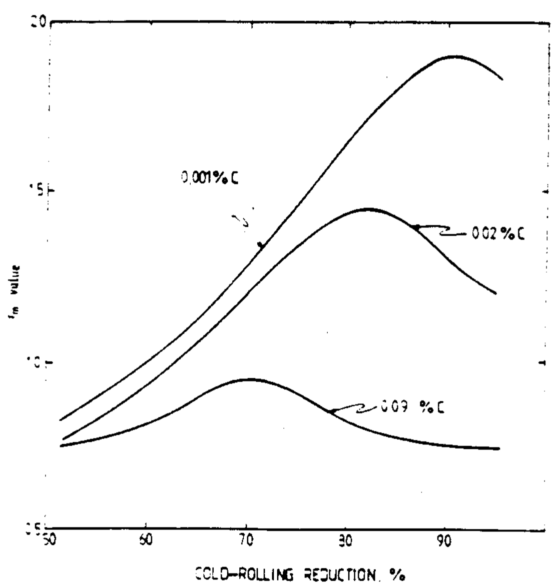


Fig. 3 - Effect of carbon content on r_m with varying amounts of cold-rolling in steel (from Fukada, ref. 11).

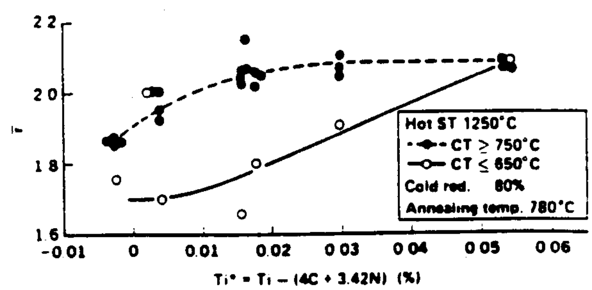


Fig. 4 - Effect of Ti*, excess Ti, and coiling temperature on r_m in Ti-IF steels (from Katoh et al., ref. 16).

TABLE 2 - Solubility Expressions for Various Precipitates
Found in HSLA and IF Steels

Compound	K_s	Reference
NbC	-2500/T - 0.63	13
NbC	-9290/T + 4.37	"
NbC	-9100/T + 3.7	"
NbC	-7290/T + 3.04	"
NbC	-7900/T + 3.3	"
$NbC_{.87}$	-7020/T + 2.81	14
$NbC_{.87}$	-9830/T + 4.33	"
Nb(C+N)	-5860/T + 1.54	13
Nb(C+12/14N)	-6770/T + 2.26	"
$NbC_{.87}$	-9800/T + 4.46	"
$NbC_{.24}N_{.65}$	-7520/T + 3.11	"
NbN	-10400/T + 4.09	"
NbN	-10230/T + 4.04	"
NbN	-10800/T + 3.70	"
NbN	-10150/T + 3.79	14
TiC	-12170/T + 4.91	"
TiC	-7000/T + 2.75	13
TiN	-10230/T + 4.45	14
TiN	-20790/T + 2.0	13
TiN	-19740/T + 6.75	"
TiN	-8000/T + 0.322	"
TiN	-15020/T + 3.82	"
TiN	-15790/T + 5.40	14
AlN	-18420/T + 6.40	"
AlN	-6770/T + 1.033	13
AlN	-6180/T + 0.725	"
AlN	-7400/T + 1.95	"
AlN	-7750/T + 1.8	"
AlN	-7500/T + 1.48	"
AlN	-8790/T + 4.45	14

which affect texture formation related to C interactions and Mn content. Figure 7 also shows that the effect of Mn on r_m values decreases with decreasing C content and is minimal in Ti-stabilized steel (18).

A recent study by Kawasaki et al. (19) demonstrates the complexity of the interactions between Mn, S, C, and Ti which contribute to mechanical property development in Ti-containing IF steels. Figure 8 shows the effect of Mn on strength, ductility and r_m at two levels of S content, and Figure 9 shows the effect of Mn at the two S levels on aging index and bake hardening index. Aging was performed at 100°C after a prestrain of 10 pct and baking was simulated by heating at 170°C after 2 pct prestrain. The results were correlated with the identification of the precipitates formed in the various steels. Without Mn, only Ti (C,N) and $Ti_4C_2S_2$ were observed. With Mn at the 1.00 wt pct level, MnS and TiC replace $Ti_4C_2S_2$ as the precipitates which combine with S and C. As a result less carbon is combined as $Ti_4C_2S_2$, and more C is available to go into solution and contribute to aging and bake hardening.

Phosphorus

Phosphorus additions provide a very effective mechanism of imparting increased solid solution strengthening to IF steels. This increased strengthening due to P can be obtained together with high r_m values (20). However careful process and compositional control are necessary to prevent cold work embrittlement due to phosphorus segregation to ferrite grain boundaries during and after annealing (21). McMahon (22) has shown that C tends to segregate to ferrite grain boundaries in low C steel and provides very strong grain boundary bonding. In ULC steels, C is not available, and consequently P segregates to ferrite grain boundaries during annealing, resulting in a sensitivity to intergranular fracture during subsequent forming operations.

The key to controlling P embrittlement is to adjust chemistries and processing to provide just enough interstitial carbon to displace phosphorus at ferrite grain boundaries without degrading formability. Figure 10 shows the interrelationship of P content, aging index and cracking in a Nb-containing IF steel. As noted above, the aging index is a good indicator of the amount of C in interstitial solution after annealing. Figure 10 shows that the higher the aging index, implying greater amounts of C available for grain boundary bonding, the lower the sensitivity to cold work cracking even at relatively high P contents. In order to provide just the right amount of available C in P-alloyed IF steels, Ti and Nb levels should be lower than those in low P steels.

Iron-titanium-phosphide precipitates may form in high-P, Ti-containing IF steels. Irie et al. (21) indicate that coarse FeTiP particles may enhance the nucleation of recrystallized ferrite grains with random orientation and thereby decrease {111} components of texture. However, other studies have shown that control of FeTiP precipitation may enhance {111} texture formation. Okamoto and Mizui (23) have analyzed the complex sequence of precipitate formation as a function of temperature and composition in IF steels. Figure 11 shows schematic diagrams of these precipitate sequences in two types of steel and shows that FeTiP precipitates form at the lowest temperatures. According to the precipitation sequences shown, FeTiP precipitation

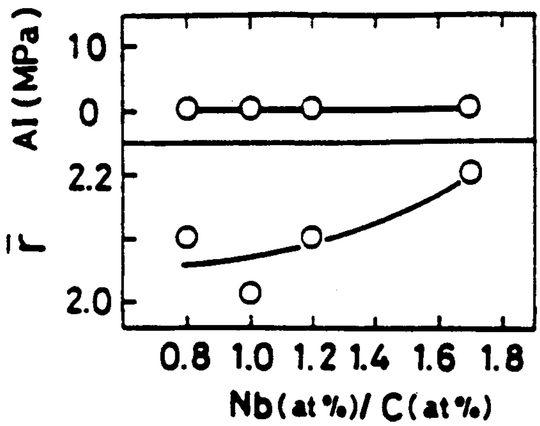


Fig. 5 - Effect of Atomic ratio of Nb/C on the aging index, AI, and r_m of Nb-stabilized IF steel with C=40 ppm, continuously annealed at 830°C (from Ohashi et al., ref. 17).

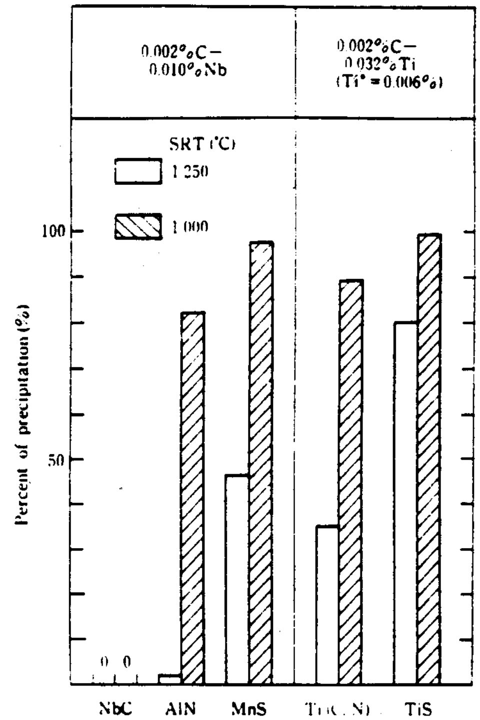


Fig. 6 - Effect of slab reheating temperature on dissolution of precipitates in Ti- and Nb-stabilized steels (from Ohashi et al., ref. 17).

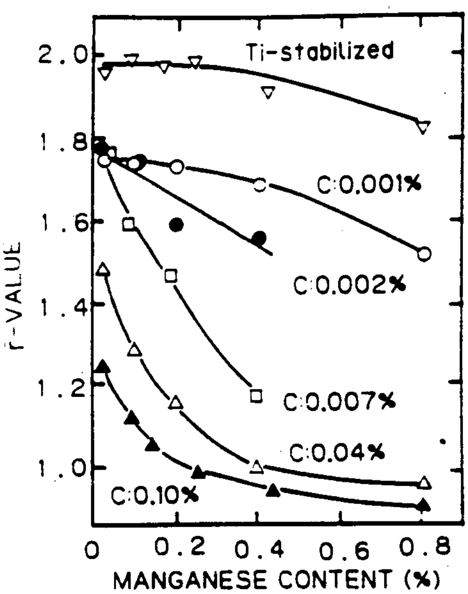


Fig. 7 - Effect of Mn content on r_m of steels with various carbon contents (from Matsudo et al., ref. 18).

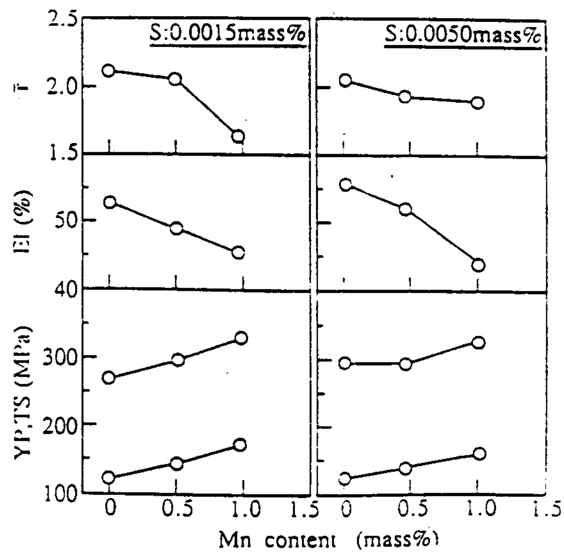


Fig. 8 - Effect of Mn and S content on mechanical properties of cold rolled and continuously annealed Ti-stabilized IF steel (from Kawasaki et al., ref. 19).

could be suppressed in hot band during low temperature coiling. The authors show that high rates of heating during recrystallization annealing prevent the precipitation of FeTiP precipitates until after the nucleation of favorable {111} grains. Subsequent precipitation of FeTiP then prevents the nucleation and growth of ferrite grains with undesirable orientations and thereby makes possible the production of annealed sheet with high r_m values.

PROCESSING FACTORS

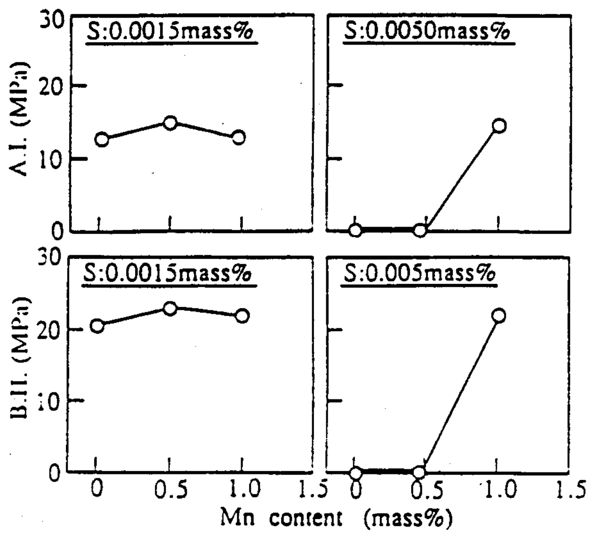
Ultra-low carbon steels are produced by several sequential processing operations which include steelmaking and vacuum degassing, casting to slabs, reheating, hot rolling to hot band or strip, coiling, cold rolling, and annealing. As noted in the discussions above, each processing step influences the microstructural evolution of cold rolled and annealed sheet of high formability.

Interstitial-free steels stabilized with Ti and Nb are ideally suited for rapid continuous annealing. Aluminum-killed deep drawing quality steels are well suited for batch annealing in which the aluminum nitride precipitation responsible for good textures is suppressed until annealing. The very slow heating rates of cold-rolled coils during batch annealing then permits AlN precipitation. Stabilized IF steels develop precipitate dispersions in the hot band prior to cold rolling and annealing (24). Therefore the precipitates are in place prior to annealing and the control of texture occurs even during the high rates of heating characteristic of continuous annealing lines.

The very low interstitial content of annealed ferrite microstructures of IF steels obviates the need for overaging steps following the recrystallization stage of continuous annealing. In higher C sheet steels overaging after continuous annealing is necessary to precipitate carbides and reduce the tendency to aging which creates Luders bands and reduced ductility (25). Although annealed IF steels show continuous yielding after annealing, low-strain temper rolling step is usually performed to control shape and surface texture (15).

The stabilizing elements in IF steels drastically reduce recrystallization rates of cold rolled sheet and annealing temperatures around 700°C (12,26). However, the increased temperature range of ferrite stability in IF steels makes possible annealing in the single phase ferrite field. At much higher temperatures then can be used for higher carbon steels. Thus temperatures between 800 and 850°C are typically used for continuous annealing, and at these temperature even with stabilizing element precipitate dispersions, recrystallization is fully accomplished within minutes. Osawa et al. (26), Figure 12, show that higher temperature annealing of Ti-stabilized ELC steels produces higher r_m values than does lower temperature annealing. At low annealing temperatures microstructures may not be completely recrystallized within the time span of continuous annealing, and at the highest annealing temperatures, around 900°C, longer soaking times cause precipitate coarsening, ferrite grain growth, and remarkable increases in r_m values.

The stabilizing element precipitates which combine with carbon, i.e. $Ti_4C_2S_2$, TiC, and NbC, are largely produced by control of slab reheating, hot rolling, and coiling. The earlier discussion on the effects of Ti and Nb has indicated that Ti compounds tend to precipitate



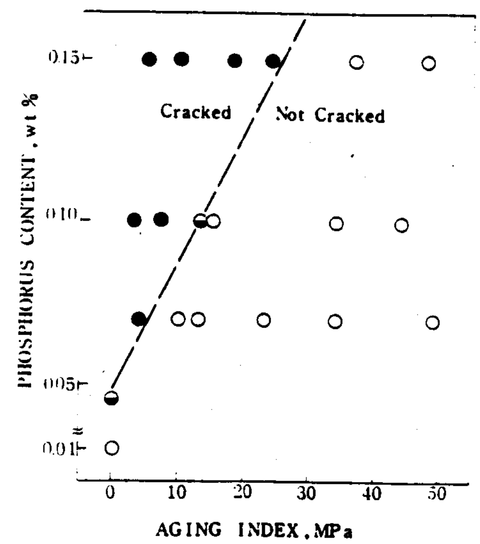
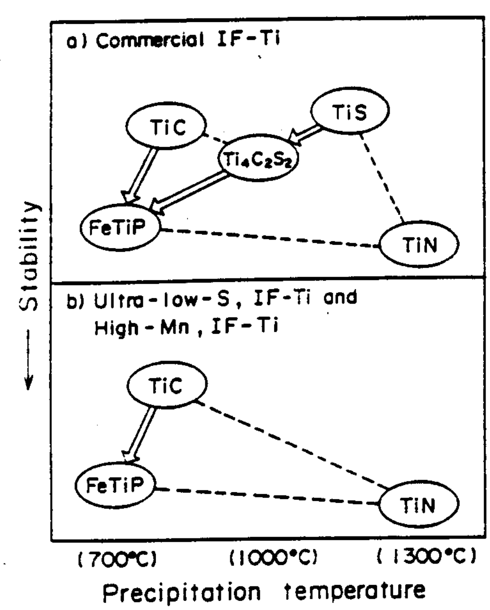


Fig. 9 - Effect of Mn and S content on aging index (AI), and bake hardenability (BH), of cold rolled and continuously annealed Ti-stabilized IF steel (from Kawasaki et al., ref. 19).

Fig. 10 - Effect of aging index and P content on cold-embrittlement (T_{test}=0°C) in a Nb stabilized IF steel (from Irie et al., ref. 21).



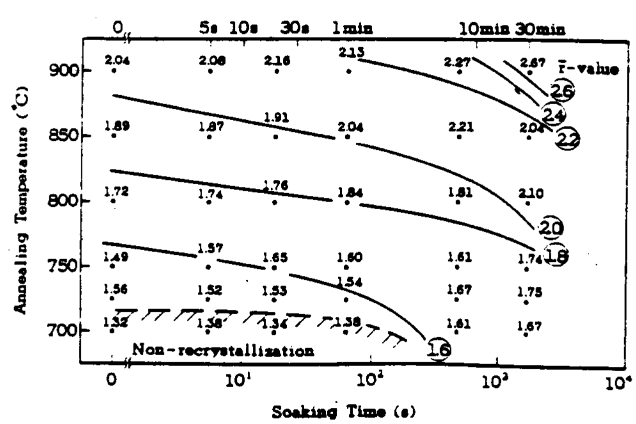


Fig. 11 - Schematic representation of precipitation temperature and stability of Ti precipitates in Ti-stabilized IF steels (from Okamoto et al., ref. 23).

Fig. 12 - Effect of continuous annealing soaking temperature and time on r_m (Osawa et al., ref. 26).

at higher temperatures than do Nb compounds. Work by Jonas, Yue, and their colleagues (27-29) confirms differences in hot rolling IF steel behavior which are consistent with differences in Ti and Nb precipitation. They found that the flow stresses of both types of IF steel increase with decreasing deformation temperature in the austenite range, and then drop significantly if hot deformation is continued into the range of ferrite stability. The flow stresses and strain hardening of a Nb-containing steel, however, were significantly higher than those of a Ti-containing steel during low-temperature austenite deformation. Finer ferrite grain sizes were also observed in Nb-containing IF steel finished at low temperatures. These observations are consistent with strain-induced Nb(C,N) precipitation on dislocation substructure and suppression of recrystallization of austenite deformed at low finishing temperatures in higher carbon steels (30). In contrast, the occurrence of precipitation of Ti(C,N) and $Ti_4C_2S_2$ in a Ti-stabilized IF steel did not significantly influence austenite recrystallization during hot deformation (27).

The effectiveness of stabilizing alloying element additions in removing C from interstitial solid solution produces cold rolled and annealed sheet of high ductility and formability but with low strength. Table 3 shows typical properties. The low strengths increase sensitivity to denting in vehicles, and therefore, active areas of development include strengthening approaches such as solid solution strengthening with phosphorus and bake hardening as described earlier. These approaches require the best of alloying and processing control and are fruitful areas of development.

TABLE 3 - Typical Properties of IF DDQ Steels (from ref. 4,5,6)

YS MPa	YS MPa	El	n	$\bar{r}$	Δr
140-180	290-340	40 50	0.22 0.31	1.8 2.2	-0.08-0.9

SUMMARY

This overview of ultralow carbon steels has briefly described some of the factors which contribute to their microstructural evolution and high formability. The development of these steels truly represents an outstanding achievement of the integration of processing and physical metallurgical approaches to alloy and process design. The presentation has relied heavily on previous reviews and symposia (4,5,6), and was designed to provide a current overview rather than an exhaustive literature survey.

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