Basic studies leading to the development of an ultrahigh strength, high fracture toughness low-alloy steel

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Abstract. Ultrahigh strength steels have been used increasingly in recent years for critical aircraft and aerospace structural applications. In such applications, though materials performance is of prime consideration, cost and availability makes the low-alloy steels an attractive option. This paper describes the development of an ultrahigh strength NiSiCrCoMo low-alloy steel, supported by significant findings obtained from the basic studies that were aimed at understanding how solute additions influence fracture resistance of iron, with and without the presence of carbon. The results of the basic studies, in combination with the work of Garrison (1986) on a NiSiCr steel, have profitably been employed in the development of a NiSiCrCoMo low-alloy steel possessing a strength–toughness combination quite comparable to the highly alloyed 250-grade maraging steel. Reproducibility of attractive strength and toughness properties has been established in tonnage scale melts. This steel, in the softened condition, has good formability and machinability. Weld parameters have also been established. The NiSiCrCoMo low-alloy steel thus meets the requirements of performance and cost rendering it an attractive option for advanced structural applications.

Keywords. Ultrahigh strength low-alloy steel; fracture toughness; alloy softening; tempered martensite embrittlement.

1. Introduction

Steels for several high-technology applications, such as aircraft and aerospace, need to possess ultrahigh strength coupled with high fracture toughness in order to meet the requirement of minimum weight while ensuring high reliability. In such applications, though materials performance is of prime consideration, cost and availability makes the low-alloy steels an attractive option. The commercial use of low-alloy steels for such applications has often been limited by their low fracture toughness. It is well-known that increase in strength is generally accompanied by a decrease in toughness. Considerable research effort has been directed over the past two decades or so towards improving fracture toughness of low-alloy steels at yield strengths $\geqslant 1400$ MPa. As recently reviewed by Tomita (1991), these efforts can broadly be classified into three methods, namely (i) thermal and thermomechanical treatments, (ii) control of sulphide inclusions and (iii) design of new alloys. Here we present the work carried out by us aimed at development of a low-alloy NiSiCrCoMo steel possessing strength–toughness combination quite comparable to highly alloyed 250-grade maraging steel.

We embarked upon a research programme with the aim of understanding the fracture toughness behaviour of iron and its alloys, which constitute the basis for widely used engineering steels. Evaluation of fracture behaviour of iron-based solid solutions, with cobalt, molybdenum, nickel or silicon as solutes, was undertaken. To establish the influence of alloying additions in the presence of carbon, the studies were extended to Fe–C–X alloys. The understanding thus developed, in combination with

the work of Garrison (1986) on a NiSiCr steel, has been profitably employed for the development of a NiSiCrCoMo low-alloy steel.

2. Basic studies

Solute additions to metals to form solid solutions invariably results in improvement in strength with a concomitant decrease in fracture resistance. There exists a fair amount of understanding as to how solutes influence strength of the alloys (Leslie 1972; Pickering 1978). However, understanding in regard to resistance to fracture, in comparable depth, is yet to evolve. To-date studies pertaining to the fracture resistance of iron and its solid solutions are limited and have been carried out in terms of either tensile ductility or impact transition temperature, which parameters do not always correlate well with the fracture toughness. In our work, the J-integral method was used for the measurement of ductile fracture toughness J_{IC} of eight solid solution alloys of Armco iron containing cobalt, molybdenum, nickel or silicon at two levels of concentration. The studies were then extended to Fe–C based alloys.

2.1 *Solid solution alloys of iron*

Solid solution alloys of iron with Co, Mo or Ni at 0·5 and 5·0 wt.% and Si at 0·5 and 3·5 wt.% concentrations were processed, the details of which are given elsewhere (Srinivas 1991). Armco iron, Fe–0·5 Co, Fe–5 Co and Fe–0·5 Mo exhibited stable crack extension while Fe–0·5 Ni, Fe–5 Ni, Fe–0·5 Si, Fe–3·5 Si and Fe–5 Mo showed cleavage instability during loading of precracked fracture toughness specimens (Srinivas *et al* 1994a). J–R curve technique as well as critical stretch zone width were employed to derive J_{IC} in the former case while in the latter case the value of J at the onset of cleavage instability was taken as J_{Q} and subjected to validity checks. Fracture toughness J_{IC} data for the four binary alloys are presented in table 1 and plotted as a function of alloying content in

Table 1. Tensile, impact and fracture toughness properties for Armco iron and its binary alloys.

Material	Grain size ($\bar{L}/\mu$m)	Yield stress (MPa)	UTS (MPa)	n	CVN impact energy at RT (J)	DBTT* (K)	J_{IC} (kJ/m^{-2})	Notch yield ratio**
Armco iron	78	189	296	0·29	250	243	170	—
Armco iron	118	180	296	0·28	260	251	140	2·5
Fe–0·5 Si	125	225	385	0·21	8	330	97	—
Fe–3·5 Si	140	423	541	0·15	6	405	42	1·5
Fe–0·5 Mo	125	187	359	0·27	52	276	112	—
Fe–5·0 Mo	120	212	385	0·22	40	285	102	—
Fe–0·5 Ni	125	195	335	0·25	8	353	59	—
Fe–5·0 Ni	80	257	386	0·21	15	423	80	—
Fe–0·5 Co	130	110	290	0·30	210	253	162	—
Fe–5·0 Co	125	130	293	0·35	270	263	187	3·7

*20 J (15 ft. lb) criterion
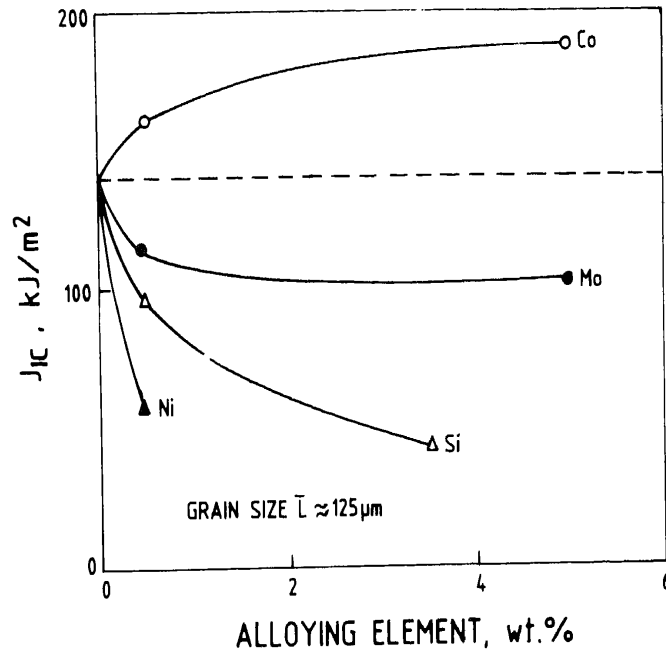**Ratio of notch tensile strength and yield strength

Figure 1. Influence of solute additions on fracture toughness of Armco iron at a constant grain size of 125 μm.

figure 1. The notable observation is that cobalt imparts to Armco iron significant improvement in fracture toughness. On the other hand, Si and Ni have an increasing degree of deleterious effect on Armco iron in that order, with the least effect being noticed with Mo (Srinivas *et al* 1991a, 1994a).

The large decrease in J_{IC} with silicon and higher molybdenum concentration is explained on the basis of a change in fracture mode from ductile to cleavage as a result of stress concentration ahead of the crack tip reaching the cleavage fracture stress. On the other hand, the loss in fracture toughness with nickel addition is attributed to sulphur segregation at grain boundaries which results in pockets of intergranular fracture.

Mo, Ni or Si increases the yield strength (YS) of iron. On the other hand, cobalt lowers the yield strength of iron, with no observable effect on ultimate tensile strength (UTS) (table 1). The extent of reduction in yield strength (alloy softening) was seen to decrease with increasing cobalt content. Through secondary ion mass spectroscopy (SIMS), experimental evidence has been obtained, for the first time, for the suggestion that the 'scavenging' of interstitials (Rees *et al* 1951; Stoloff *et al* 1965; Ravi and Gibala 1969; Smialek *et al* 1970; Pink *et al* 1980; Ishii *et al* 1984) leads to alloy softening. Ion images for Armco iron, figures 2a and 2b, clearly indicate that the interstitial carbon is homogeneously distributed in the solution. On the other hand, C^- and CN^- ion images for Fe–5 Co alloy, figures 2c and 2d, clearly reveal segregation of carbon and nitrogen at the grain boundaries. It was also observed through yield stress dependence on grain size that the Hall-Petch constants σ_0 and k_y substantiate SIMS observations (Srinivas *et al* 1994a).

The increase in J_{IC} of Armco iron due to cobalt addition is primarily attributable to the increase in the strain hardening exponent n. The critical plastic zone size increases with increasing n. Furthermore, increase in n results in slip dispersal and reduces the void growth rate. Increased n as a result of cobalt addition is therefore expected to delay void nucleation and decrease the void growth rate. It thus appears that cobalt addition toughens iron by influencing the energy spent in the plastic zone formation as well as in the subsequent stages of the ductile fracture process, namely void nucleation, growth and coalescence of ductile fracture. If the lowering of yield strength
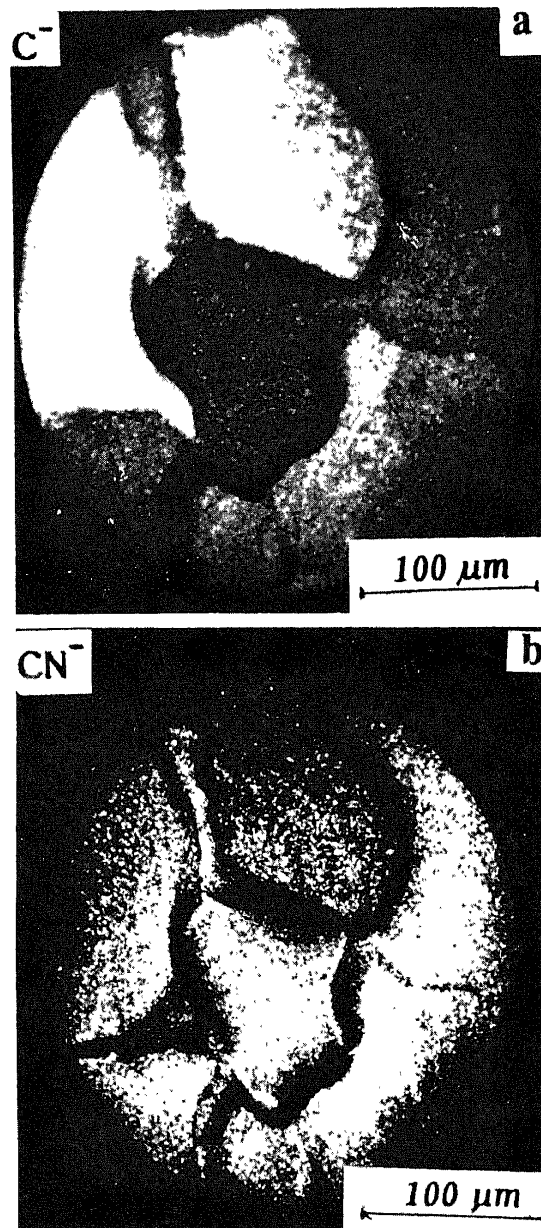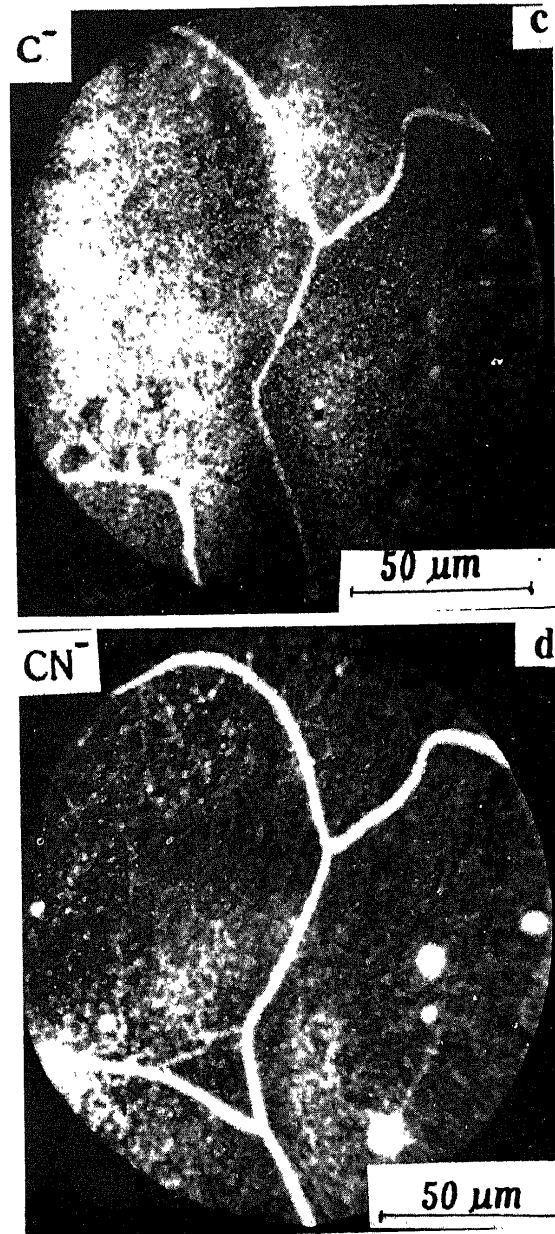
Figure 2a–b.

Figure 2. SIMS ion images showing (a, b) uniform distribution of interstitials in Armco iron, and (c, d) scavenging of interstitials to grain boundaries in Fe–5 Co alloy.

by cobalt addition to iron were to be the prime reason for the enhancement of J_{IC}, addition of 0·5% Co, which reduced the yield strength by 40% as compared with a 25% decrease with 5% Co addition, should have resulted in a larger increase in J_{IC}. Contrary to these expectations, a 5% cobalt addition caused a higher increase in J_{IC} of iron (35%). Although Fe–Co alloys have lower yield strengths than Armco iron, the flow stress builds up rapidly as a result of higher strain hardening rate at the early stages of plastic deformation.

A new approach has been proposed (Srinivas *et al* 1994b) to the calculation of J_{IC} in terms of the critical strain criterion model, originally developed by Rice and Johnson (1970) and Ritchie and Thompson (1985), for engineering alloys having a substantial volume fraction of second phase particles. The sequence of events leading to crack initiation is assumed to be as follows. As soon as the pre-cracked sample is loaded in tension under a monotonically increasing load, extensive blunting of the crack tip occurs. The crack blunting process is completed, leading to a constant stretch zone width (SZW), before the void nucleation and growth occur at a characteristic distance l_c from the blunted crack tip. The mechanism of void nucleation ahead of the blunted crack and also the characteristic distance l_c of the void from the crack tip have been determined for a single phase material, for the first time, through a careful examination of the interrupted fracture toughness test specimens (Srinivas 1991; Srinivas *et al* 1994b). The proposed approach not only incorporates the experimentally measured l_c values but also the plastic flow related energy dissipation in the Hutchinson–Rice–Rosengren zone beyond the process zone. Theoretically predicted J_{IC} values are compared in figure 3 with those derived experimentally. Figure 3 also includes data on Armco iron and nickel derived over the temperature range 298–673 K (Srinivas *et al* 1991a). It is clear
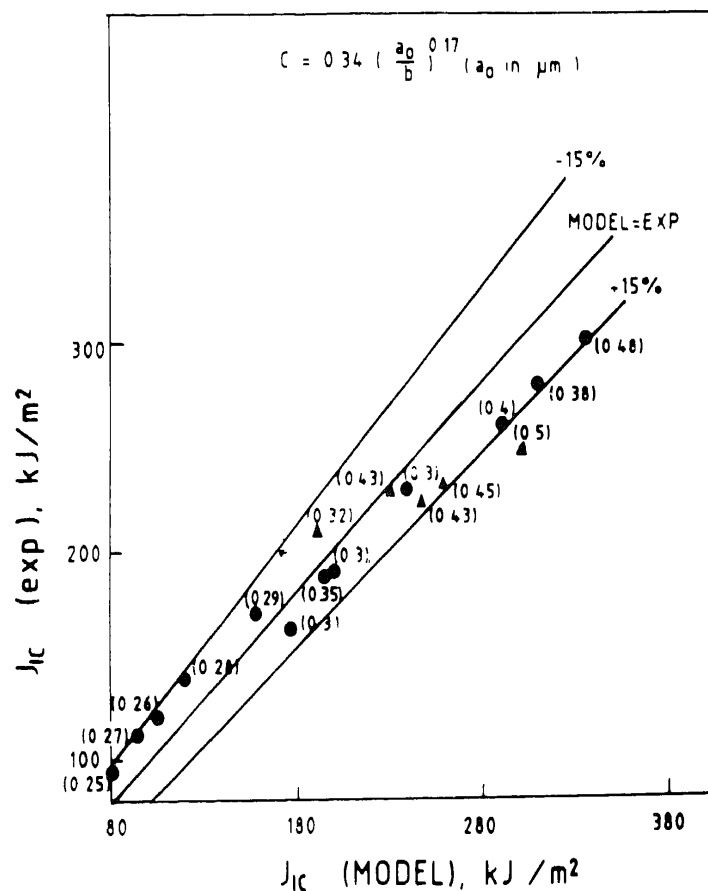

Figure 3. A comparison between experimentally measured and model predicted J_{IC} values.

from figure 3 that the proposed method estimates reasonably well (to within $\pm 15\%$) the ductile initiation fracture toughness J_{IC} of single phase materials exhibiting a wide range of values $(100-300 \text{ kJ/m}^2)$.

2.2 Fe–C–X alloys

The studies on Fe–C–X alloys have significantly revealed that the beneficial effect of cobalt on J_{IC} of iron is even more pronounced in the presence of carbon (Srinivas *et al* 1991b). The strength and toughness properties of Fe–0·2 C, Fe–0·2 C–5 Co and Fe–0·2 C–5 Ni alloys are compared in table 2. A 5% cobalt addition does not influence significantly the strength behaviour of Fe–0·2 C alloy. On the other hand, a 5% nickel addition appreciably increases the strength and reduces ductility and work hardening exponent. The addition of 5% cobalt significantly enhances (80%) J_{IC} of Fe–0·2 C alloy, while the addition of 5% nickel increases the same marginally (18%). Nickel, generally considered to be a beneficial element in the presence of carbon is seen to influence dramatically the impact toughness with only a marginal effect on J_{IC}. On the other hand, cobalt addition, with no observable effect on impact toughness, causes significant improvement in J_{IC}. These results further suggest that there is no one-to-one correlation between impact toughness and static fracture toughness for this class of alloys.

Similar findings have been reported in recent years (Ritchie *et al* 1976; Lin *et al* 1986) although, on the other hand, there is a fairly widespread use of correlations between impact toughness and fracture toughness. High temperature austenitizing treatment at 1473 K results in a two-fold increase in fracture toughness K_{IC} of AISI 4340 steel but gives rise to inferior charpy impact energy as compared to conventional 1143 K austenitizing treatment (Ritchie *et al* 1976). This behaviour was shown to be independent of shear lip energy and the variation in strain rate imposed by K_{IC} and impact tests. Such an effect was found to result from the differing response of each microstructure in regard to the influence of notch root radius on toughness. Ritchie *et al* (1976) proposed that failure occurs when the maximum tensile stress exceeds the critical fracture stress σ_F over a characteristic distance ahead of a sharp crack (K_{IC}) or at the elastic-plastic interface ahead of a rounded notch (impact). Fracture toughness of 1473 K structure is higher because the characteristic distance (associated with coarser grain size) is larger while the higher impact toughness of 1143 K structure is a result of larger fracture stress. On the other hand, the reasons for the dramatic effect of nickel on impact toughness observed in the present study could be traced to alloy softening at high strain rates prevailing under impact conditions (Srinivas *et al* 1991c),

Table 2. Tensile, impact and fracture toughness properties of Fe–C–X alloys.

Material	YS (MPa)	UTS (MPa)	El. (25 mm GL) (%)	CVN impact energy (J)	J_{IC} (kJ/m^2)
Fe–0·2 C	244	370	44	16	130
Fe–0·2 C–5 Co	242	409	47	15	232
Fe–0·2 C–5 Ni	297	467	37	120	153

as also suggested by Jolley (1968) and Petch (1987). The microstructural features, namely ferrite grain size and the volume fraction of pearlite were maintained nearly the same in all the three Fe–C based alloys under consideration.

3. Development of NiSiCrCoMo steel

It is evident from the foregoing that cobalt toughens iron, and even more positively in the presence of carbon. In our efforts to arrive at a new alloy chemistry that possesses improved fracture toughness at ultrahigh strength levels, cobalt addition was made to Garrison NiSiCr steel.

3.1 *Cobalt modified NiSiCr steels*

Silicon is a solid solution strengthening element. It is known to delay tempering and shift tempered martensite embrittlement to higher temperatures, which features enable retention of ultrahigh strength levels even after tempering at relatively higher temperatures. Silicon is also known to influence sulphide distribution in the presence of nickel (Garrison 1986). Silicon refines sulphide particle size and increases sulphide spacing and thereby causes an increase in fracture toughness by rendering the fracture
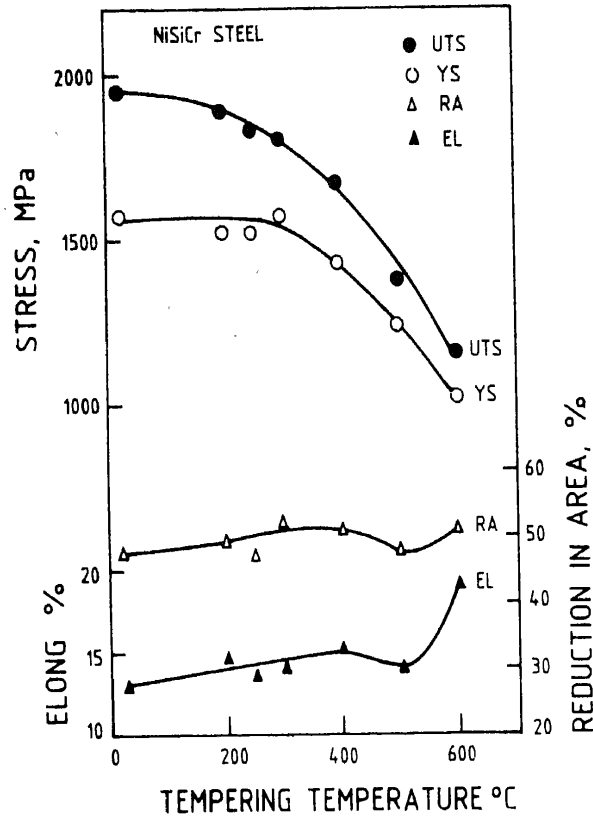

Figure 4. Tensile properties of NiSiCr steel as a function of tempering temperature.

process that involves void nucleation, growth and linkage more difficult. In view of these observations, Garrison (1986) NiSiCr steel containing high silicon has been, chosen as the base material.

The variation of strength with tempering temperature (figure 4) shows that the NiSiCr steel retains high strength up to 250° C. Beyond about 300° C, there occurs a steep fall in strength, which is also accompanied by a loss in ductility. A trough in ductility variation with tempering temperature occurs in the temperature range 400 to 600° C. The impact energy at room temperature increases with tempering temperature up to around 300° C, beyond which a steep fall occurs, with a minimum at 500° C (figure 5). It is evident from the data presented in figures 4 and 5 that the steel develops an optimum combination of strength and impact toughness in the 250° C tempered condition. Accordingly, fracture toughness and ductile-to-brittle transition temperature (DBTT) were evaluated for the steel in the 250° C tempered condition. Fracture toughness tests conducted according to ASTM standard E399 yielded K_{IC} of 103 MPa$\sqrt{m}$. The impact toughness in the 250° C tempered condition is plotted against test temperature in figure 6. The 20 J criterion yields a DBTT of $-100°$ C. Mechanical properties of the base Garrison NiSiCr steel subjected to an optimum treatment of 250° C tempering are summarized in table 3.

Transmission electron microscopy (TEM) and Auger electron spectroscopy (AES) studies have revealed (Malakondaiah *et al* 1994) that the loss in ductility and impact toughness of NiSiCr steel in the tempering temperature range 350 to 600° C is due to the simultaneous occurrence of tempered martensite embrittlement (TME) and tempered embrittlement (TE). It is now well established that TME occurs in low-alloy
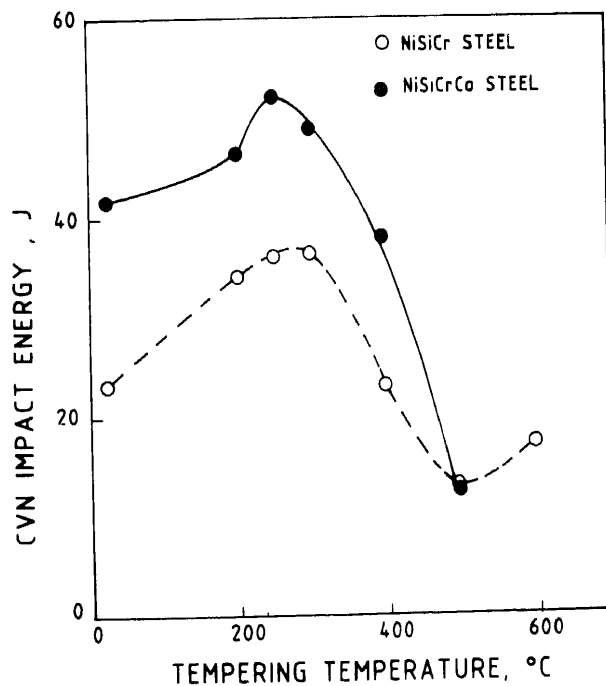

Figure 5. Charpy V-notch impact energy as a function of tempering temperature for NiSiCr and NiSiCrCo steels.
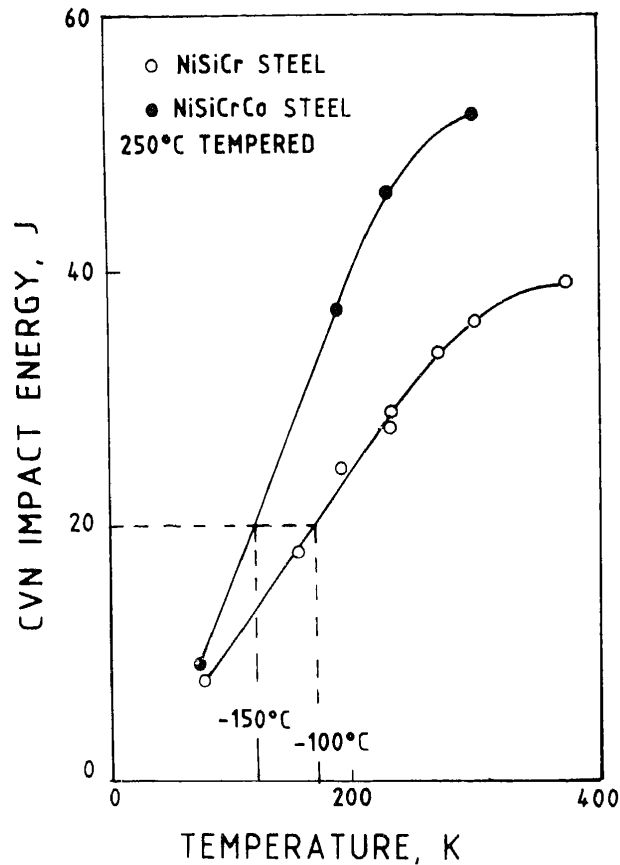
Figure 6. Variation of Charpy V-notch impact energy with test temperature for NiSiCr and NiSiCrCo steels in 250°C tempered condition.

Table 3. Mechanical properties of steels developed in the present study in the optimum heat treated condition.

Material	YS (MPa)	UTS (MPa)	RA (%)	CVN impact energy at RT (J)	K_{IC} (MPa$\sqrt{m}$)
NiSiCr	1550	1825	47	36	103
NiSiCrCo	1360	1670	50	52	140
NiSiCrMo	1648	1904	48	35	102
NiSiCrCoMo	1530	1890	48	34	120

steels when tempered in the temperature range 250 to 400° C. A higher temperature range of 350 to 600° C for NiSiCr steel is understandable as silicon is known to shift TME to higher temperatures. TEM carried out on 250, 500 and 600° C tempered specimens revealed the presence of interlath-retained austenite in the 250° C tempered condition, transformation of retained austenite to carbides in the 500° C tempered condition and coarse interlath carbides in the 600° C tempered condition (figure 7).
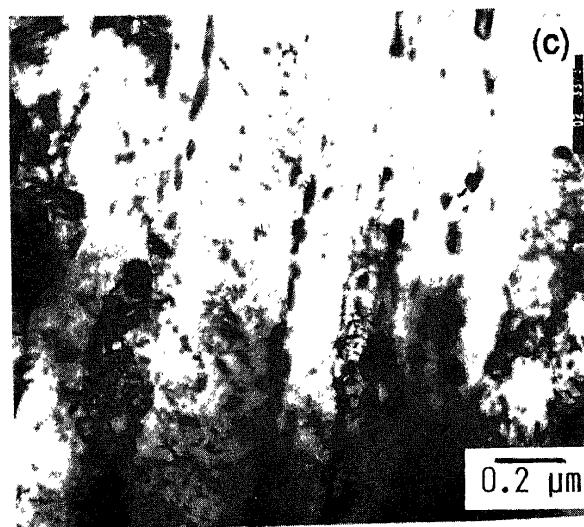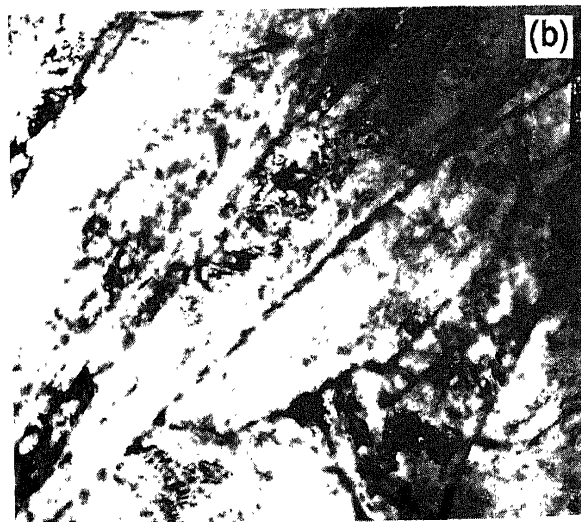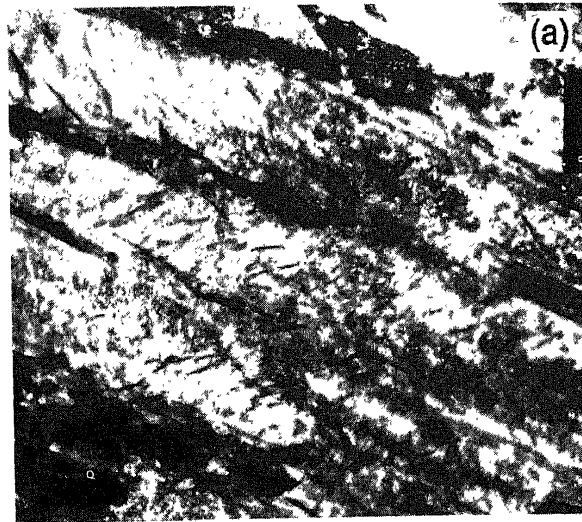
Figure 7. TEM micrographs of NiSiCr steel tempered at (a) 250°C and (b) 500°C showing interlath retained austenite and carbide stringers respectively. (c) Coarse interlath carbides are seen in the 600°C tempered condition.

Further, AES carried out on 500° C tempered condition revealed segregation of phosphorous, which was absent in the as-quenched condition, suggesting occurrence of temper embrittlement.

Cobalt addition was made to the base NiSiCr steel to achieve further improvements in fracture toughness. The tempering behaviour of NiSiCrCo steel, shown in figure 8, reveals that the steel retains higher strength up to 300° C, as against 250° C observed with NiSiCr steel. Ductility shows a trough in the tempering temperature range 400 to 600° C. The impact energy data included in figure 5 reveals that the impact toughness increases with tempering temperature up to 250° C, beyond which a steep fall occurs. It is evident from figures 5 and 8 that NiSiCrCo steel, in a manner similar to that observed with the base NiSiCr steel, develops an optimum combination of strength and impact toughness in the 250° C tempered condition. Fracture toughness tests carried out in this optimum condition yielded a K_{IC} of 140 MPa$\sqrt{m}$. The 20 J criterion yields a DBTT of $-150°$ C (figure 6). Mechanical properties of NiSiCrCo steel in 250° C tempered condition are included in table 3. A significant increase in fracture toughness is seen with accompanying loss in strength. However, the loss in strength is not in proportion to the increase in fracture toughness. A 40% increase in fracture toughness is seen while the loss in strength is around 10%. More than 40% increase in impact toughness is also evident (table 3).

The increase in fracture toughness with an accompanying decrement in strength with cobalt addition to base steel is in line with the observations made on cobalt
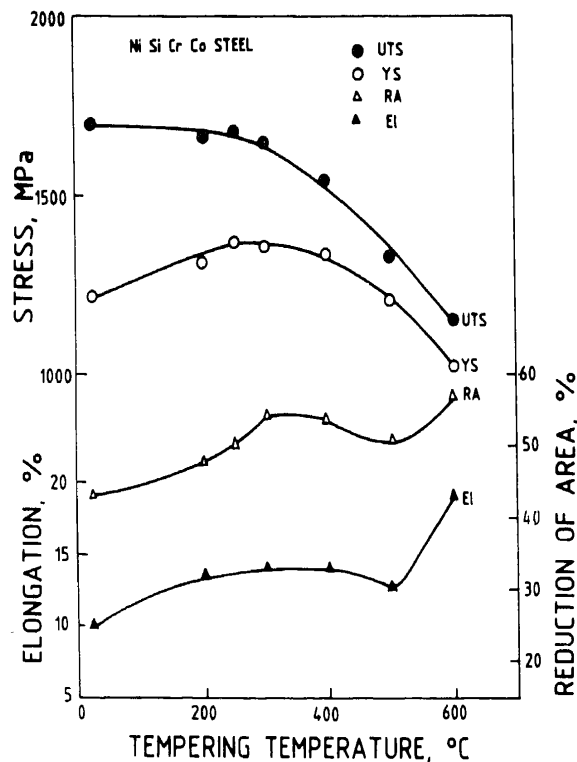

Figure 8. Tensile properties of NiSiCrCo steel as a function of tempering temperature.
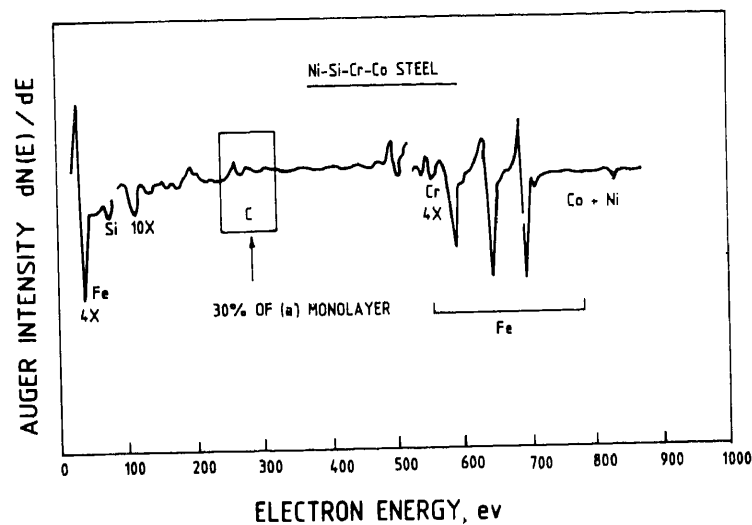
Figure 9. Scanning Auger spectrum showing carbon segregation at grain boundaries in NiSiCrCo steel.

additions to Armco iron (Srinivas *et al* 1994a). Microsegregation studies carried out on NiSiCrCo steel using a scanning auger microscope show segregation of carbon to the grain boundaries (figure 9). The increase in fracture toughness with cobalt addition is attributable to the segregation of carbon to the grain boundaries, thus increasing the cohesive strength of the grain boundary and rendering the crack initiation process much more difficult (Olson 1990; Misra and Rama Rao 1993).

To build up strength in the presence of cobalt in the base NiSiCr steel, molybdenum addition was resorted to. The studies on iron based solid solutions have shown that, while molybdenum is a solid solution strengthener, the fracture toughness of Armco iron is only marginally lowered as a result of molybdenum addition, below a certain level of concentration. The influence of molybdenum on the base NiSiCr steel was therefore studied. The study has interestingly revealed that the addition of molybdenum to the base NiSiCr steel leads to an increase in yield and ultimate tensile strength with negligible reduction in fracture toughness (table 3). The increase in yield strength is as much as 7% while the increase in UTS is around 5%.

As a follow-up, molybdenum addition was made to the cobalt containing steel. To optimize the composition further, melts with varying content of Co and Mo were taken and processed. The tempering behaviour of each steel was studied and the optimum property combination was established. The fracture toughness of the optimized NiSiCrCoMo steel is nearly 15% higher than that of the base Garrison NiSiCr steel at a matching strength level (table 3). The transmission electron micrograph corresponding to the optimum condition, shown in figure 10, shows lightly tempered lath martensitic structure. SIMS studies carried out on NiSiCrCoMo steel in the optimized condition pointed to carbon segregation to grain boundaries (figure 11) reinforcing the suggestion that grain boundary carbon contributes to enhanced fracture toughness, observed in the results on binary Fe–Co alloys, even when the steel is a complex composition of six alloying elements.
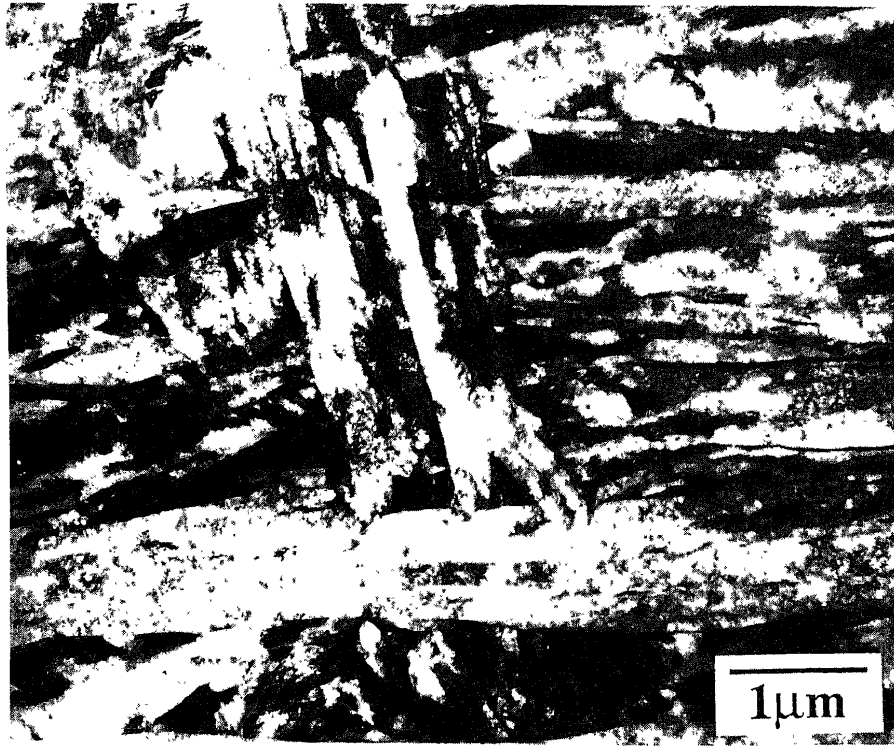
Figure 10. TEM micrograph of NiSiCrCoMo steel showing lightly tempered lath martensitic structure in the optimum heat treated condition.

3.2 Comparison with other ultrahigh strength steels

The properties of NiSiCr, NiSiCrCo, NiSiCrMo and NiSiCrCoMo steels are compared in figure 12 with those of the low-alloy steels AISI 4340, 15CDV6 and D6ac, and highly alloyed HP 9–4–30 and maraging steels. Figure 12 shows the range of properties attainable with low-alloy quenched and tempered steels and 18 Ni maraging steels as two separate bands. Among the three low-alloy steels under consideration, namely 15CDV6, AISI 4340 and D6ac, the properties of D6ac steel that was developed for aircraft and missile structural applications fall in the maraging steel regime. While the strength–toughness combination of the base Garrison NiSiCr steel is quite comparable to that of D6ac steel, the cobalt and molybdenum containing NiSiCrCoMo steel possesses properties superior to those of D6ac steel. The NiSiCrCoMo steel is also superior to HP 9–4–30 in terms of strength. The strength–toughness data for NiSiCrCo and NiSiCrCoMo steels fall in the upper bound range for 250-grade maraging steel, with a total alloying content in excess of 30%.

3.3 Reproducibility of properties of NiSiCrCoMo steel on tonnage scale

Two 5 ton capacity melts of NiSiCrCoMo steel were taken and processed by Mishra Dhatu Nigam Limited (MIDHANI), a special alloy undertaking. Electric arc melting, followed by vacuum arc refining process, was employed. The mechanical properties of

Figure 11. SIMS ion image showing carbon segregation to grain boundaries in NiSiCrCoMo steel.
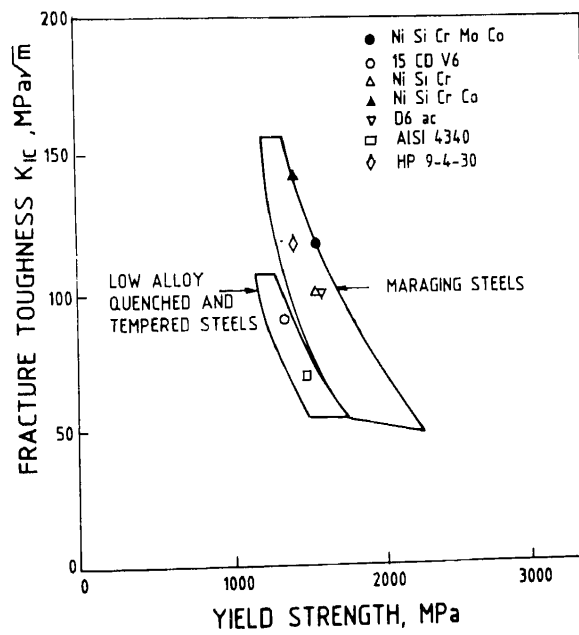

Figure 12. Fracture toughness vs yield strength for various ultrahigh strength steels.

Table 4. Mechanical properties of NiSiCrCoMo steel.

Property	Lab-scale melt	Industrial-scale melt
YS (MPa)	1530	1610
UTS (MPa)	1890	1970
El. (25 mm GL) (%)	14	13·6
CVN impact energy at RT (J)	34	28–32*
K_{IC} (MPa$\sqrt{m}$)	120	90–110*

*The property range is due to the properties obtained at various stages of processing of the ingot to hot-formed products.

Table 5. A comparison of mechanical properties in the softened condition.

Steel	YS (MPa)	UTS (MPa)	El. (%) (25 mm GL)
NiSiCrCoMo (740° C/4h, Furnace cooled)	652	853	19·4
250-grade Maraging (Solution treated)	725	1000	18·0

NiSiCrCoMo steel processed on this scale are compared in table 4 with those derived from a laboratory-scale melt. The attractive combination of strength and toughness observed in the laboratory melt is seen to have been reproduced in the industrial-scale melt.

3.4 Machinability and formability

Spheroidise-annealing (740° C) softens the NiSiCrCoMo steel considerably. The properties of the steel in the spheroidise-annealed condition are, again, quite comparable to those of the 250-grade maraging steel in the solutionized condition (table 5). NiSiCrCoMo steel in the softened condition can easily be rough machined before finally subjecting to hardening and tempering treatment in order to develop an optimum combination of strength and fracture toughness.

The steel in the softened condition also possesses good formability. Erichsen cupping test yielded a cup depth of 10·8 mm for 2·3 mm sheet. In conformity with these observations, industrial trials of cold forming revealed that a 7 mm thick blank of 570 mm diameter could be formed, without intermediate annealing, to a shell of 350 mm inner radius.

3.5 Weldability

Gas tungsten-arc (GTA) process, generally recommended for low-alloy steels, has been chosen. Detailed studies were undertaken to arrive at a suitable filler material. Tension test results of butt-welded and heat-treated 2·3 mm thick sheet specimens revealed that the W2 filler (trade name of MIDHANI for the filler developed for

250-grade maraging steel) yields 96% weld efficiency in terms of yield strength. Experiments have shown that the base metal strength could be realized in the butt-welded and heat-treated condition with base steel as filler. Efforts are now underway to process the base steel in the form of wires for use as filler.

The NiSiCrCoMo steel discussed in this presentation promises to be an inexpensive, and therefore attractive, substitute for expensive, high alloy steel like the well-known maraging steel for light-weight, high performance structural applications.

Acknowledgements

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