Intergranular Failure in Micro-alloyed Steels and its Relationship to Carbon Content

B. MINTZ and Z. MOHAMED
Department of Mechanical Engineering, The City University, London EC1V 0HB, UK

ABSTRACT

The influence of C on the hot ductility of C-Mn-Al and C-Mn-Al-Nb steels has been examined after solution treatment and cooling to test temperatures in the range 700° to 1100°C and testing at a strain rate of 3 x $10^{-3}s^{-1}$. Raising the C level from 0.06% to 0.15% in a C-Mn-Al steel was found to move the ductility trough to lower temperatures and the movements were similar to those expected from the change in Ae_3 temperature. As no AlN was precipitated under the conditions of cooling and strain rate used in the tests the hot ductility behaviour was regarded as being the same as that for plain C-Mn steels. It was therefore concluded that the fall in ductility that occurs after solution treating and cooling to the test temperature was due to strain concentration in thin films of deformation induced ferrite, producing voiding round inclusions which gradually link up to give intergranular failure. The position of the trough is probably determined by the Ae_3 and Ar_3 temperature, ductility improving again when the temperature falls so that there is sufficient transformation induced ferrite present to prevent strain concentration. Introducing Nb into the steel widened the trough. Raising the C level in the C-Mn-Nb-Al steels from 0.014% to 0.16%C did not influence the position of the trough but the depth increased with C level. Quite a marked deterioration in hot ductility occurred on raising the C level from 0.014% to 0.1%C but a further increase to 0.16%C produced only a small change. The widening of the trough was due to NbCN precipitation and quite good agreement was obtained between the degree of NbCN precipitated, as calculated from the solubility data and the Reduction of Area values. At the lower temperature side of the ductility trough both precipitation and deformation induced ferrite were present.

KEYWORDS

Intergranular Failure, Hot Ductility, HSLA Steels.

INTRODUCTION

Intergranular failure has been found to be very common in steels tensile tested in the temperature range 700° to 1000°C at low strain rates (3 x $10^{-3}s^{-1}$), (Mintz and Arrowsmith [1979], Ouchi and Matsumoto [1982]).

Micro-alloyed steels give very low ductilities as measured by Reduction of Area (R of A), and even plain C-Mn steels exhibit this form of failure if the strain rate is sufficiently low, (Crowther and Mintz [1986a]).

In the case of the ductility trough obtained on cooling from the austenite, the mechanism proposed for intergranular failure has been linked to the onset of transformation from the austenite to ferrite. It is suggested, (Crowther and Mintz [1986a]), that thin films of the softer ferrite phase form round the γ grain boundaries and strain concentration occurs leading to intergranular failure. Because C has such a pronounced influence on the temperature for transformation, Crowther and Mintz [1986a] investigated the hot ductility of plain C-Mn steels having a range of C contents in the temperature range 700° to 1000°C after first heating to 1330°C to produce a coarse γ grain size. Raising the C content from 0.04% to 0.28%C caused the trough to move to lower temperatures in agreement with the observed changes in transformation temperature. Failure was by strain concentration in the softer ferrite phase enveloping the γ grains, causing void formation at MnS inclusions which gradually link up to give intergranular cracks. The present examination is an extension of this work to the influence of carbon on the hot ductility of micro-alloyed steels.

EXPERIMENTAL

C-Mn-Al and C-Mn-Al-Nb steels were chosen for examination. The range of C examined in the C-Mn-Al steel was .056 to 0.15% C and for the C-Mn-Al-Nb steel 0.014% to .16% C. The full compositions wt.per cent of the Nb and Al containing steels are given in the table below.

Steel	C	Mn	Si	S	P	N	Al	Nb
C-Mn-Al	.056	1.46	.42	.012	.007	.007	.034	-
"	.11	1.42	.32	.002	.011	.0038	.038	-
"	.15	1.45	.29	.008	.003	.006	.017	-
C-Mn-Al-Nb	.014	1.48	.36	.006	.015	.007	.033	.028
"	.10	1.39	.42	.010	.007	.0075	.036	.026
"	.16	1.24	.22	.005	.011	.009	.028	.023

The steels were supplied as hot rolled 12 mm plate. Tensile samples having a length of 70 mm and diameter 7.93 mm were machined from the plates with their axis parallel to the rolling direction. Hot ductility tensile tests were carried out on a Hounsfield Tensometer and heating was supplied by an induction heater. The samples were protected from oxidation by an Argon atmosphere.

Samples were heated in 15 min to 1330°C held 5 min. prior to cooling to test temperatures in the range 750° - 1000°C at an average rate of 60°C min^{-1}. Samples were held for 5 min at test temperature before straining to failure at 3 x $10^{-3}s^{-1}$.

Fracture surfaces were examined using a Jeol T100 SEM. Carbon extraction replicas were prepared from transverse sections approximately 1 mm behind the fracture surface, and examined using a Jeol 100 kV, TEM.

To establish γ grain size prior to fracture, small samples were heated in a muffle furnace to simulate the thermal cycles of the hot tensile test, and then cooled at a rate to produce outlining by ferrite of the austenite grains. The linear intercept method was used to measure the austenite grain size.

RESULTS

Hot Ductility Curves

The curves of R of A against test temperature for the Al and Nb containing steels with different carbon contents are shown in Figures 1 and 2 respectively. Ae_3 temperatures calculated from the formula of Andrews [1965] are marked on the curves.

All the steels show a marked ductility trough with different depths, widths and positions dependent on the steel compositions.

For the Al containing steels, Figure 1, the depth and width of the troughs were very similar. Minimum ductilities were in the range 47 to 50% R of A. The major effect of increasing the carbon content was to shift the hot ductility curve to lower temperatures. Raising the C level from 0.056% to 0.15% C caused the ductility trough to shift by approximately 70°C.

For the Nb containing steels, raising the C level increased both the depth of the trough and its width. However, the position of the minimum ductility temperature did not change with increase in carbon level, this being constant at 800°C. For the temperature range in excess of 800°C differences in hot ductility between the 0.1 and 0.16% C steel were small, the higher C steel having ~5% lower R of A. Lowering the C level to 0.014% gave a large improvement in hot ductility (~25% increase in R of A being observed).

Examination of the stress-total elongation curves for the C-Mn-Al steels showed that the temperature for dynamic recrystallisation was 900°C for all the carbon levels examined.

For the Nb containing steels the temperature for dynamic recrystallisation increased with C level being 950°C for the steel with 0.014%C and between 1000° and 1100°C for the steel with 0.16%C.

SEM Fractography and Metallography

SEM fractography revealed three distinct fracture modes:
a) High temperature ductile rupture (HTDR) observed in all steels for R of A values ≥90%, Figure 3a Wray [1981] has observed this mode of failure in austenitic iron. In the present work, large voids are apparent on the fracture surface and apparently not associated with second phase particles. Crowther and Mintz [1986a] showed the same observations in plain carbon steels and in micro-alloyed steels, (Crowther and Mintz [1986b]) for R of A values greater than 80%. They believed that the large voids apparent on the fracture surface were originally intergranular cracks, formed at an early stage of deformation. As deformation proceeds, the original grain boundary crack is distorted into an elongated void, until final failure occurs by necking between these voids.

b) Intergranular micro-void coalescence (IMC). This failure mode was observed at the lower R of A values, Figure 3b. Micro-voids, often associated with second phase particles, cover the facets of the austenite grains. Analyses of second phase particles under the TEM shows that these particles are MnS or MnFeS inclusions.

c) Intergranular decohesion (ID) was also often observed at the lower ductility values particularly for the Nb containing steels, Figure 3c. This mode of failure is distinguished from IMC by flat austenite grain facets, which, although showing MnS inclusions, lack micro-voiding. These ID failures were similar to those observed by Crowther and Mintz [1986b], Ouchi and Matsumoto [1982] and Maehara and Ohmori [1984] and are believed to be due to grain boundary sliding. Frequently fractures were a mixture of IMC and ID with the tendency for the proportion of IMC to increase as the temperature was lowered below the Ae_3.

Transformation took place too rapidly to quench in deformation induced ferrite in the C-Mn-Al steels but some evidence for it was found in the C-Mn-Al-Nb steel, Figure 4.

No AlN precipitation was found in C-Mn-Al samples fractured at their minimum ductility temperatures. However extensive NbCN precipitation both at prior γ grain boundaries and within the matrix was found in the 0.16% and 0.1% C steels, Figure 5a. NbCN precipitation was sparse in the 0.014% C steels, Figure 5b.

No significant influence of C content on γ grain size could be discerned although the γ grain size of the Nb containing steel was coarser than that of the C-Mn-Al steels (490 μm and 280 μm respectively).

DISCUSSION

Hot Ductility behaviour of C-Mn-Al steel on raising the C level

The precipitation of AlN after solution treatment has been shown to be very sluggish, (Gladman and Pickering [1967]). Very high concentrations of Al and N are required before AlN is able to precipitate out[8]. It has been shown for example, Crowther et al., [1987], that for the 0.15% C steel having 0.017% soluble Al and 0.006% N, no precipitation of Al occurred after holding for six hours at 850°C, the temperature corresponding to that for the maximum rate of precipitation. Dynamic precipitation at these concentrations has also not been observed, (Crowther et al., [1987]). Michel and Jonas [1981] have shown that in order to obtain dynamic precipitation of AlN at a temperature of 875°C the solubility product [Al].[N] has to be $\geqslant 6.8 \times 10^{-4}$. It is therefore not surprising that no precipitation of AlN has been observed in samples tested in the temperature range 750° to 850°C, the temperature range of low ductility and intergranular fracture.

It is therefore reasonable to assume that the behaviour for the C-Mn-Al steel should be identical to that shown by the previously examined plain C-Mn steels, (Crowther and Mintz [1986a]). In the latter case the ductility trough was shown to be transformation controlled. The failure mode at the minimum ductility temperature was along thin films of ferrite which formed round the austenite grains generally by deformation induced transformation. The softer ferrite allowed strain concentration to occur causing micro-void coalescence at MnS inclusions, with the voids eventually linking to give intergranular failure. With coarse grained material as in the present case, (grain size ~280 μm), it has been shown, (Crowther and Mintz [1986a]) that deformation raises the Ar_3 temperature to the Ae_3 and therefore the initial fall in hot ductility should correspond approximately to the Ae_3 temperature and the shift in the hot ductility curve should be related to the change in the Ae_3 temperature.

The present findings are in reasonable accord with this in that the initial fall in hot ductility corresponds approximately to the Ae_3 temperature and the change in the Ae_3 temperature on raising the C level from 0.056% to 0.16%C is 45°C compared to the observed change of 70°C. Subsequent recovery of ductility at temperatures just below the Ar_3 has been shown,

(Crowther and Mintz [1986a]) to take place by thickening of the ferrite films surrounding the γ grains and/or a reduction in the relative difference in strength between the austenite and ferrite phases. The position of the trough can therefore be defined by the Ar_3 temperature on the low temperature side and the Ae_3 on the high temperature side.

The present results are in agreement with those of Hannerz [1985] who showed that increasing the carbon content from 0.06% to 0.28% in a C-Mn-Al steel improved hot ductility in the temperature range 750 to 900°C and Suzuki et al's, [1984] results who found similar improvements on increasing the C level from 0.05% to 0.4% C in a silicon and manganese free steel containing aluminium additions.

Hot Ductility Behaviour of C-Mn-Nb-Al steel on raising the C level

The major difference between the C-Mn-Al steels and the C-Mn-Nb-Al steel was the deepening and widening of the trough particularly at the higher C levels (0.1 to 0.16% C). Such behaviour, (Mintz and Arrowsmith [1980]) has been shown to be due to precipitation of NbCN. The overriding importance of precipitation in controlling the hot ductility in these steels might also be deduced from the observation that the minimum ductility temperature, 800°C, remains the same with C content. Dynamic precipitation is very marked in Nb containing steels after solution treatment and their very poor hot ductility is ascribed to a combination of extensive matrix and γ grain boundary precipitation and the tendency to form precipitate-free zones which lead to strain concentration at the grain boundaries. Contrary to AlN precipitation, NbCN precipitation has been shown to be very fast. Static precipitation of NbCN in an 0.16% C steel similar to the one under examination has also been shown to start after 1 s at 950°C and dynamic precipitation is extremely rapid, (Crowther et al., [1987]). Jonas and Weiss [1979] showed that dynamic precipitation can be complete in about 100 s at 900°C, the temperature corresponding to the maximum rate of precipitation which is significantly less than the time taken to complete a test (approximately 5 mins).

It therefore may be a reasonable approximation to assume that most of the precipitation obtained under equilibrium conditions is precipitated out rapidly during the test at least at temperatures close to that giving the maximum rate of precipitation. In fact the position and shape of the curve can be well explained by using equilibrium solubility data. If Irvine et al's [1967] solubility formula for NbC is used:

$$\log[\mathrm{Nb}].[\mathrm{C}] = -\frac{6770}{T} + 2.26 \qquad (1)$$

where T = the absolute temperature K, then the equilibrium volume fraction of NbC present can be calculated. If this is plotted against temperature, Figure 2, then it can be seen that there is quite a good agreement between the improvement in hot ductility with increase in temperature and the reduction in the volume fraction of NbC. The better hot ductility behaviour shown by the very low C steel (0.014% C) compared to the higher C steels can readily be explained in terms of the much lower volume fraction of precipitation present (see Figures 5a and b), and the relative insensitivity of the hot ductility to raising the C level above 0.1% can also be understood as precipitation is not greatly influenced by further changes in C level.

This relative insensitivity of hot ductility to C level in the range 0.08 to 0.2% has also been noted by Ouchi and Matusmoto [1982] in steels containing 0.03% Nb when tested in the temperature range 600° to 1000°C after solution treating at 1330°C. Maehara and Ohmori [1984] have also noted the insensitivity of hot ductility to C level in the range 0.05% to 0.3% C for steels containing 0.05% Nb when tested at 800° and 900°C after solution treating at 1300°C. Some change in hot ductility might have been expected at the lower C level examined (.05%) but it should be noted that raising the Nb content from 0.03% to 0.05% would be expected from the solubility data to increase the amount of NbC precipitated and is equivalent to raising the C level. In their higher Nb containing steel a marked improvement in hot ductility on reducing C levels below 0.3%C would only be noted when the C levels are extremely low (<0.03% C).

Although a large part of the trough for the two higher C containing steels occurs in the temperature range in which deformation takes place solely in the γ, the actual minimum ductility occurs below the Ae_3 suggesting that as well as precipitation reducing hot ductility, the strain concentration produced in the thin films of deformation induced ferrite surrounding the γ grains must also be helping to reduce hot ductility, Figure 4. Subsequent recovery of hot ductility on lowering the temperature further may be due to both a reduction in the amount of NbCN precipitated (the kinetics of precipitation are a C curve centred at 900°C, Jonas and Weiss [1979]), and a thickening of the ferrite film.

SUMMARY

Raising the C level in a C-Mn-Al steel moves the hot ductility curves to lower temperatures in accord with the change in Ae_3 temperature. Raising the C level in a Nb containing steel has little influence on the position of the trough as ductility is controlled by precipitation of NbCN.

The depth and width of the trough is also fairly insensitive to C level, except for the very low C levels when precipitation is significantly reduced so that hot ductility improves.

REFERENCES

ANDREWS, K.W. [1965] *J.Iron Steel Inst.*, 203, 721.
CROWTHER, D.N. and MINTZ, B. [1986] *Mater.Sci.Technol.*, 2, 671.
CROWTHER, D.N. and MINTZ, B. [1986] *Mater.Sci.Technol.*, 5, 1099.
CROWTHER, D.N., MOHAMED, Z. and MINTZ, B. [1987] *Metall.Trans.*, 18A ,1929.
GLADMAN, T. and PICKERING, F.B. [1967] *J.Iron Steel Inst.*, 205, 653.
HANNERZ, N.E. [1985] *Trans.Iron Steel Inst. Jpn.*, 25, 149.
IRVINE, K.J., PICKERING, F.B. and GLADMAN, T. [1967] *J.Iron Steel Inst.*, 205,
JONAS, J.J. and WEISS, I. [1979] *Met.Sci.Journ.*, 13, 238.
MAEHARA, Y. and OHMORI, Y. [1984] *Mater.Sci.Eng.*, 62, 109.
MICHEL, J.P. and JONAS, J.J. [1981] *Acta Metall.*, 29, 513.
MINTZ, B. and ARROWSMITH, J.M. [1979] *Met.Technol.*, 6, 24.
MINTZ, B. and ARROWSMITH, J.M. [1980] "Hot working and forming processes (Ed. C.M. Sellars and G.J. Davies) 99, *The Metals Society*, London.
OUCHI, C. and MATSUMOTO, K. [1982] *Trans.Iron Steel Inst. Jpn.* 22, 181.
SUZUKI, H.G., NISHIMURA, S., IMAMURA, J. and NAKAMURA, Y. [1984] *Trans. Iron Steel Inst.Jpn.*, 24, 169.
WRAY, P.J. [1981] *Met.Technol.*, 8, 467.

Fig. 1. Hot ductility curves for C-Mn-Al steels

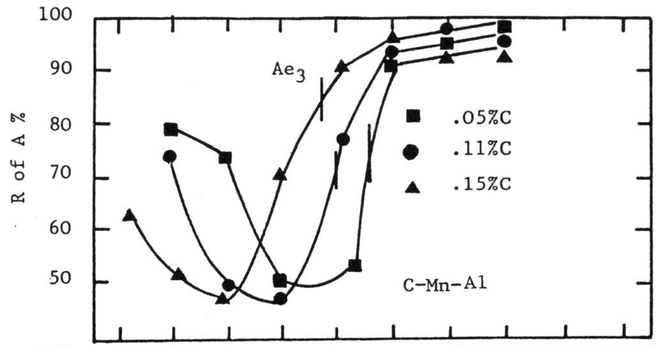
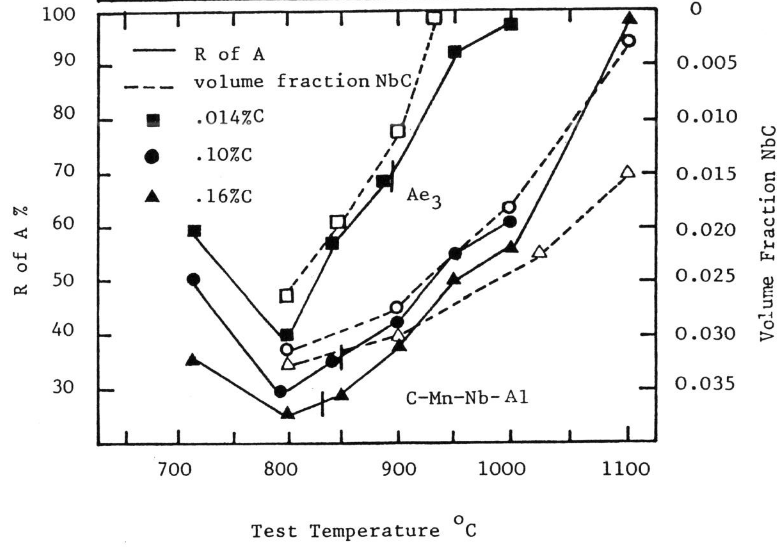
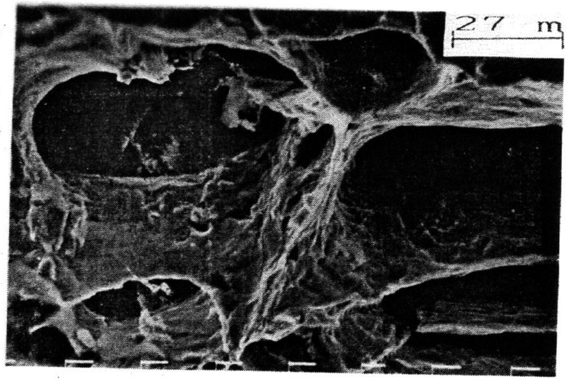
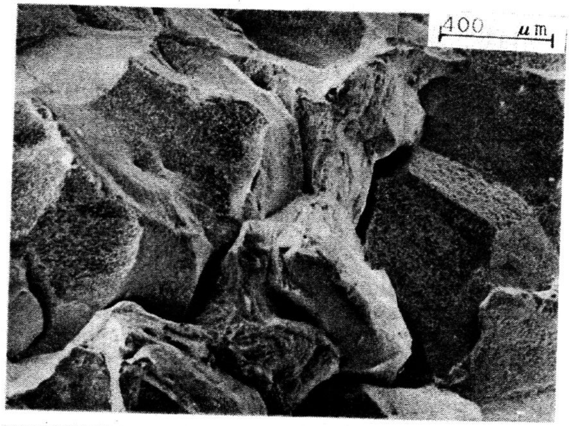


Fig. 2. Hot ductility curves for C-Mn-Nb-Al steels

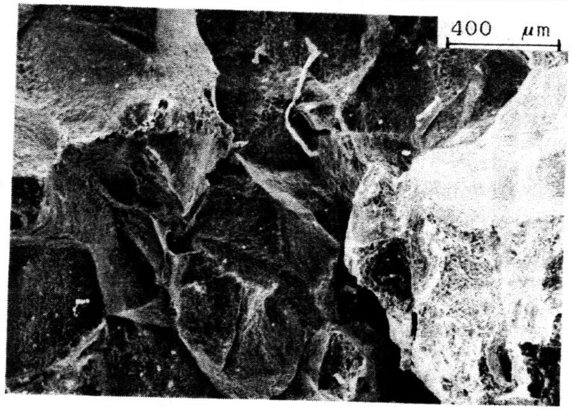




(a) High temperature ductile rupture fracture



(b) Intergranular micro-void coalescence fracture (IMC)



(c) Mixed IMC and intergranular decohesion fracture (ID)

Fig. 3. Types of fracture.

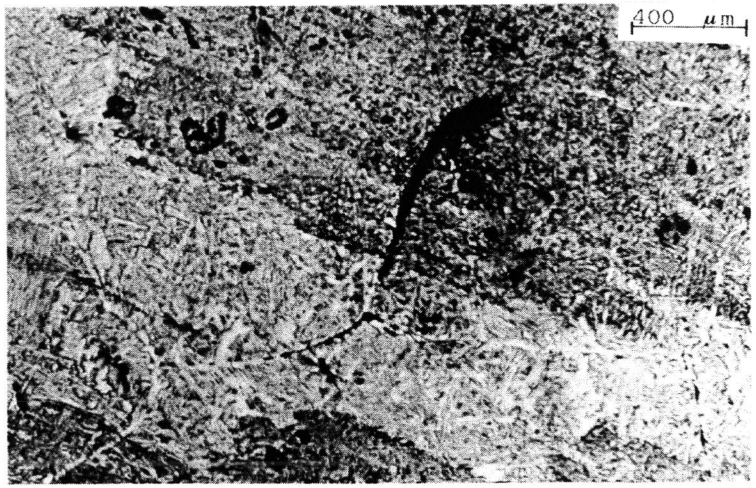


Fig. 4. 0.1% C niobium-containing steel tested at 850°C just below Ae_3 temperature

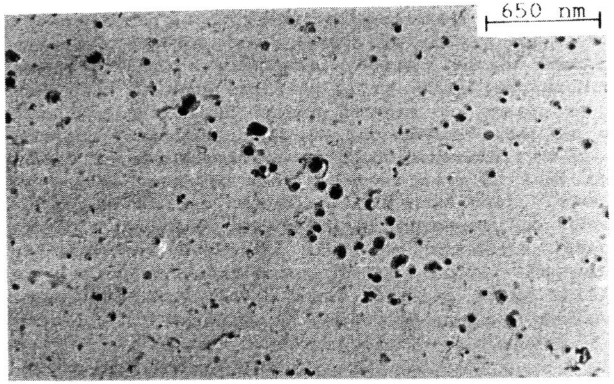


Fig. 5a. 0.1% C niobium-containing steel tested at 850°C showing extensive NbCN precipitation

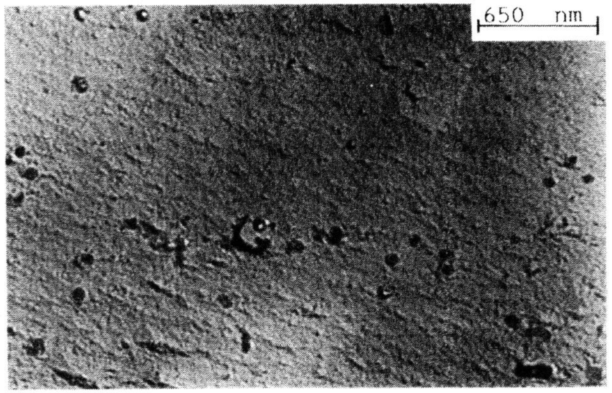


Fig. 5b. 0.014% C niobium-containing steel tested at 850°C showing sparse precipitation of NbCN