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Hot Ductility and Hot-Shortness of Steel and Measurement Techniques: A Review

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ABSTRACT

Usage of steel scrap has become important in steel making from the view points of resource conservation and preventing environmental problems. High levels of steel scraps are used in the production of steel, especially, via electric arc furnaces (EAF). However, Copper, which is mainly contained in the scrap of cars and home electronic appliance, is problematic in mechanical workings at elevated temperatures and limits the usage of scrap iron. It is enriched at steel/scale interface by preferential oxidation of Fe, which leads to liquid embrittlement or surface cracking during hot working. This type of defect is well known as surface hot shortness. Cu has been also reported to give surface cracking problem including transverse cracking in continuously cast products and has been found to be detrimental to surface quality. While nickel and silicon have been added to prevent hot shortness and cracking problem, tin, antimony and arsenic are detrimental. Silicon modifies scale of copper-bearing steels and promotes internal oxidation by the formation of 2FeO.SiO2 (fayalite). The evaluation techniques of hot shortness and cracking problem are also equally important and are mainly evaluated by hot tensile and hot bend test after oxidation. In addition to this, hot tensile test has been also used for evaluating hot ductility behavior and surface cracks of continuously cast products.

Surface Hot Shortness of Steel

Two forms of hot shortness are categorised by Melford[1], (i) liquation cracking, i.e., hot shortness in the bulk of the steel and (ii) surface hot shortness. Liquation cracking is a problem associated with enriched liquid phase at grain boundaries, which occurs when relatively insoluble residual elements (e.g. sculpture) are present in steel resulting in cracking. Surface hot shortness, on the other hand is caused by the enrichment of residual elements on the subsurface during oxidation which is liquefied and subsequently, penetrated into austenite grain boundaries. The focus of this work is surface hot shortness and therefore, no further attention will be given to liquation cracking.

Residual Elements in Steels

During steelmaking practice residual (or tramp) elements are metallic or metalloid elements that are not efficiently removed from the liquid metal and as a result may build up to relatively high levels with continued recycling.[2] Even the presence of a few hundredths to a few tenths of a percent of residual elements will have significant effect on particular critical properties of steel.[2] Elements such as Cu, Ni, Sn, Sb, Pb, As, S and P remain in steel because they cannot be preferentially oxidised during normal steelmaking processes.[1, 3] This can be understood using Ellingham diagram shown in Figure 1, in which the Gibbs free energies (in units of kJ per mole O2) of the relevant metal oxides are plotted as a function of temperature. When a steel billet/slab is reheated in an oxidising atmosphere, iron will oxidise to form a scale consisting primarily of FeO while copper and other elements whose oxides are less stable than FeO (i.e. oxides which are above FeO in the Ellingham diagram) will not oxidise. These elements will be enriched in the subsurface region of the steel. This build up adversely affects steel properties.[2]

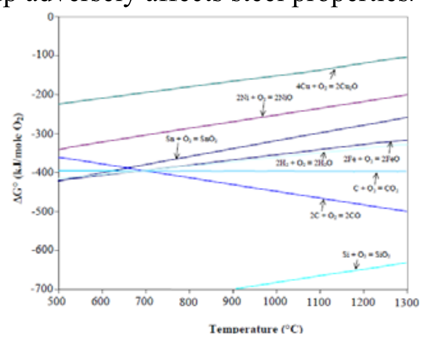


Figure.1. Ellingham diagram for various oxides.[1]

Mechanism of hot shortness

The basic mechanism of surface hot shortness of Cu bearing steel is well known.(4-6) When a Cu bearing steel is oxidised, FeO is formed due to its relatively high stability.(4,5) As discussed above, the preferential oxidation of iron leads to enrichment of such as Cu, Ni, Sn, Sb, Pb, As, S and P at the steel/scale interface. When the enrichment of copper reaches a level exceeding the solubility limit in iron, a copper-rich phase precipitates at the scale/metal interface. The iron-copper phase diagram (Figure 2) indicates that the solubility of copper in austenite (γ) at 1096°C is 8.2 wt%. Melford(1) has reported that when only copper is present in steel, an enrichment level of over 9 wt% copper is quite possible under conditions of severe oxidation in the temperature range 1100-1200°C. This enriched liquefied phase will penetrate to the austenite grain boundary resulting in cracking.

However, two contradictory views exist for hot surface cracking mechanism. One group of researchers have proposed that an applied stress is required for the material to be embrittled by the formation of a liquid copper-rich phase.(8-9) According to this group, a liquid copper-rich phase penetrates grain boundaries when the material is subject to tensile stresses. The other group of researchers have proposed that a liquid copper-rich phase penetrates grain boundaries during oxidation, i.e. in the absence of an applied stress.10-11 In the subsequent operation, i.e., during rolling the generated tensile stresses cause cracking. According to this group, cracks are formed in the reheating furnace and surface break-up occurs during hot rolling. They assume that defects are introduced into the material before hot rolling as pre-material defects.

Factors affecting hot shortness

The ease with which a copper-rich phase first precipitates at the scale/metal interface depends on the copper content in the steel, the solubility limit of copper in austenite, oxidation rate, the rate of copper diffusion and the rate of copper 'back-diffusion' from the surface to the interior.(12) The rate of oxidation and back-diffusion are both diffusion controlled mechanisms and thus affected by temperature. The reported limiting copper content below which defects arising from surface hot shortness will not occur is commonly cited as 0.2 wt% Cu.(12-13) although values of 0.15 and 0.3 wt% have also been reported. The surface cracking tendency on hot bend test specimen surfaces indicate that the severity increases with increasing copper levels.(14)

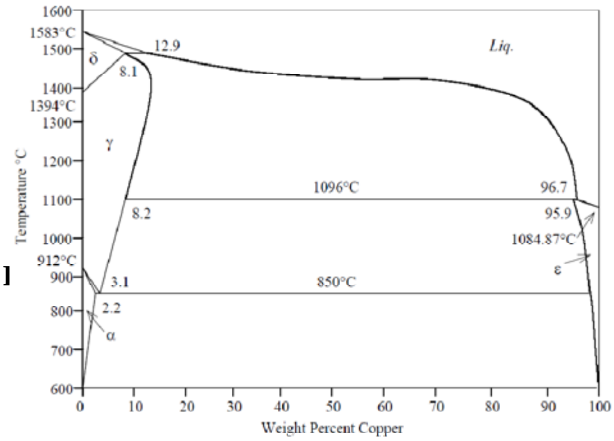


Figure 2.. Iron-copper phase diagram.(7)

The melting point of the copper-rich phase predicted from the iron-copper phase diagram is 1096°C (Figure 2) and therefore, the surface hot shortness may be expected approximately above 1100°C. In other words, no hot shortness problem will occur if the deformation temperature lower than the melting temperature of Cu. More frequently, hot shortness is observed in an intermediate temperature range of approximately 1100 - 1150°C. In this temperature range the copper-rich phase may penetrate metal grain boundaries, leading to their embrittlement during hot working. For temperatures above approximately 1200°C, the copper-rich phase is molten but mainly occluded into the scale layer. Nicholson and Murray.(15) have proposed that occlusion (or separation into the scale) occurs when internal oxides coalesce and join with the external scale, leading to engulfment of the enriched metal surface and copper-rich phase. Surface hot shortness is avoided in this situation since the molten copper-rich phase is isolated from the substrate. Figure 3 shows that internal oxides formed in 0.46Cu-0.22Ni steels after oxidised for 15 minutes at 1150°C.(16)

In contrary to the above discussion, the melting point of Cu is affected by the presence of other elements. A copper-rich phase may remain molten at temperatures as low as 900°C when Sn is present .(1-17) Sn is also a ferrite stabiliser, which will reduce the solubility of Cu in austenite.

Addition of nickel to a copper-bearing steel reduces the severity of cracking.(18-19) Ni increases the melting point of the copper-rich phase(15) apart from its beneficial effect of stabilising the austenite and increasing the solubility of copper in austenite, and thus, contributes to reducing cracking.

While Fisher[19] has pointed out that Ni : Cu ratios of 1.5-2.0 may be required to increase the solubility of Cu in austenite sufficiently to prevent the formation of liquid copper-rich phase, ratios of 1:1 or less can be effective by promoting internal oxidation and subsurface occlusion for temperatures as low as 1150°C. As reported by Kohsaka and Ouchi,[12] the critical Ni : Cu ratio for preventing surface cracking depends on the reheat temperature. The the occlusion mechanism is more pronounced at higher temperatures (i.e. where oxidation is more rapid), and therefore requires less nickel may be required to inhibit cracking at higher temperatures. Fukagawa and Fujikawa[20] has reported that a low carbon 0.50% Cu-0.023%Ni steel oxidised in air at 1250°C or higher temperatures for 2 hours did not show any cracking while no surface cracking was observed at any temperature between 1100-13000C for the 0.50%Cu-0.25%Ni steel. A copper-rich phase was observed for oxidation temperatures between 1100-1200°C of 0.50%Cu-0.023%Ni steel while the less copper-rich phase was observed above 1200°C at the scale/metal interface. Fukagawa and Fujikawa[20] proposed that the copper-rich phase becomes occluded into the scale above 12000C. This inhibited surface cracking due to the markedly decrease in quantity of copper-rich phase at the metal/scale interface.

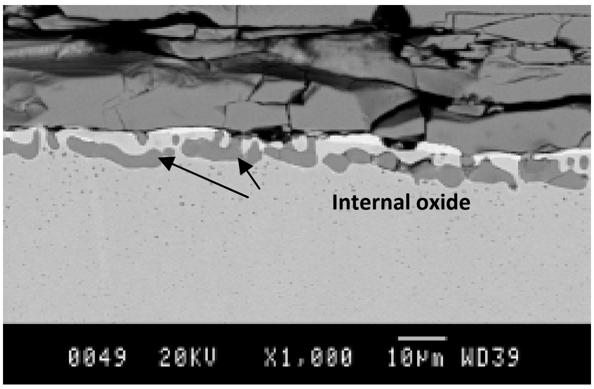


Figure 3. Appearance of internal oxides formed in 0.46Cu-0.22Ni steels after oxidised for 15 minutes at 1150°C[16]

Similar to Ni, Si has been also found to be an effective alloying element in suppressing surface hot shortness cracking.[21-22, 5] Oxidation mechanism of Si containing Cu-bearing steels is modified by the formation of $2FeO.SiO_2$ (fayalite). Fayalite is formed from internal oxidation of the silicon in steel[22, 5] and can lead to the scale becoming partly liquefied, as fayalite has a melting temperature of 1205°C and will form a eutectic with FeO (FeO-$2FeO.SiO_2$) at 1177°C[23] and with SiO_2 ($Fe2SiO_4$-SiO_2) at 1178°C[23]. In Si containing steel, formation of solid silicon-rich oxide has been attributed to increased levels of internal oxidation, and hence occlusion of copper-rich phase into the scale.[5] In other word, the presence of a solid silicon-rich oxide phase ($Fe2SiO_4$ and/or SiO_2) at the scale/metal interface inhibits the diffusion of iron from the metal to the scale.[5-23] Both of these aspects decrease the rate of oxidation which would therefore liberate less copper rich phase and decrease surface hot shortness.

As reported by Lanteri et al.[23] the oxidation rates of binary Fe-Si alloys (silicon content up to 1.42 wt%), oxidized in 1.8%O2-N2, were found to decrease with increasing silicon content below 1177°C, while higher oxidation rates was reported above 1177°C, which was attributed to the presence of a liquid phase at the scale/metal interface. Although oxidation rate is high at higher temperature, leading to high metal loss but the surface hot shortness is minimised when a liquid subscale forms. This was analysed by Kajitani et al.[24] The authors attributed that decrease in surface hot shortness is due to increased levels of occlusion. The silicon rich liquid phase in the scale may take-up the liquid copper due to a reduction in the interfacial energy between the scale and the liquid copper as the scale was liquefied.

Longitudinal and Panel cracking

Although modern steel making process involves continuous casting of steel, over two third of world steel production currently follows the conventional ingot casting route, which will continue to be an important mode of steel production for atleast some decades to come. As shown in Figure 4,[25] the formation of panel crack is one of the defects frequently appearing in the concave panel areas on fluted or corrugated ingots. This defect is also called longitudinal surface cracking, thermal stress cracking, cooling cracking, reheating cracking, phase transformation cracking, etc.[25] The reason of panel crack is attributed to a combination of reduced intermediate temperature (600-900ºC) ductility involving the presence of AlN precipitates and stress generation due to both thermal contraction and phase transformation.[25] Some of these aspects will be reviewed in details later.

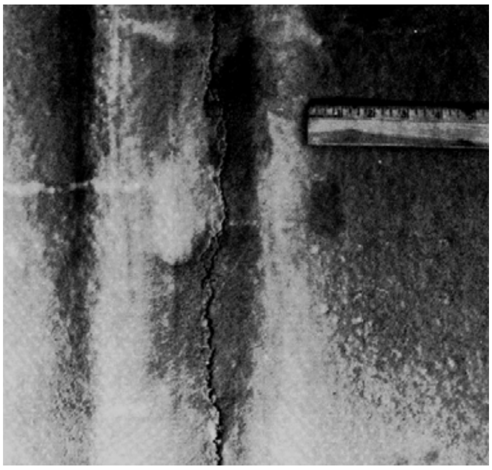


Figure 4: Typical appearance of panel crack running along corrugations of 760 x 1520 mm, 25 ton steel ingot.[25]

Transverse cracking and hot ductility

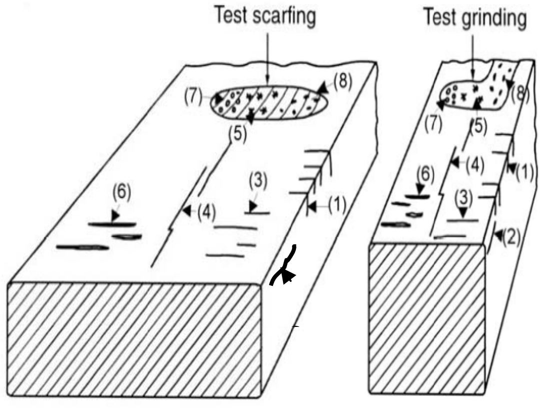
Description of Transverse Cracks

Different types of defect appeared in continuously cast products are shown schematically in Figure 4. Transverse crackings in continuous cast slabs, as reported by Harada et al.,[26] are shown in Figure 5 and 6. The most common defect found is transverse cracks, which may be formed on the broad face, narrow face, or corner of continuously cast slab. However, these are not always apparent to visual inspection unless the slab surface is scrapped or prepared for microscopic observation. These are usually associated with the depression of oscillation marks, and are predominantly found on the top slab surface. The cracks length can be several 10s of mm, and generally follow austenite grain boundaries. The cracks are partially oxidised with a little decarbonisation.[27]

Transverse cracking occurs most frequently in steels micro-alloyed with Nb, which greatly promotes the formation of transverse cracks whilet V at low N levels has no effect. However, the combinations of 0.15%V and 0.02%N have been reported to lead to transverse cracking. In copper bearing steel, Burden et al.(28) have reported that surface cracks in continuously cast blooms are associated with enrichement of Cu, Ni and Sn. The work by Woollen[29] at British steel indicates that plate steels containing Nb with additions of 0.25% Cu and 0.25% Ni have a greater incidence of transverse cracking compared with similar grades without Cu and Ni additions.

Formation mechanisms for transverse cracks

Crack formation in the material will occur when a material is unable to support an applied stress. Therefore, it is essential to understand the sources of stress during continuous casting of steel and its high temperature properties, particularly ductility.



1. Transverse corner cracks; 2. Longitudinal corner cracks; 3. Transverse cracks, 4. Longitudinal cracks (broad face); 5. Star cracks; 6. Deep oscillation marks; 7. Pinholes; 8. Macro inclusions.

Figure 4. Schematic of surface defects in continuously cast products.

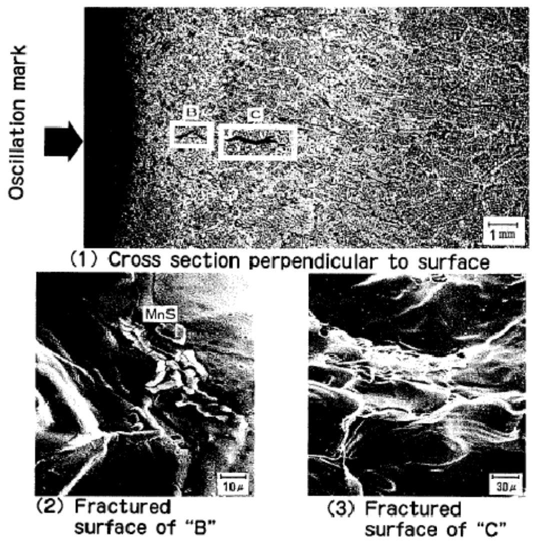


Figure 5. Fractured surface of the crack[30]

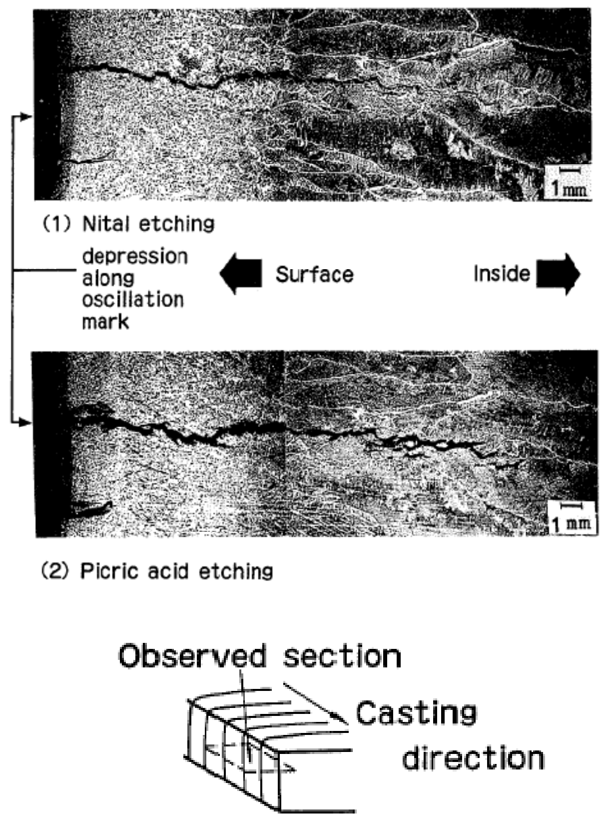


Figure 6. Large transverse crack on narrow face[30]

Lankford[30] has reviewed the source of stresses during continuous casting which can arise from a large number of different causes. These include transformation effects, thermal effects (variable heat transfer within the mould, temperature gradients within slabs, effects of cooling water sprays, contact with rollers, etc.), friction between strand and mould, bulging of the strand caused by ferrostatic pressure, mechanical effects due to misalignment of the casting machine, and straightening strains. Out of these, straightening strains is most important. This is because large transverse cracks are observed in the final straightened slab, together with the fact that these are often most numerous on the top surface of the slab (i.e. the surface which is in tension during straightening). This suggests that there is much crack propagation induced by the stresses experienced during the straightening process. When these stresses occur in the temperature range over which ductility is poor, the appearance of transverse cracking becomes severe.

Furthermore, as suggested by Harada et.al.[26] the earliest stages of transverse crack formation occur in the mould, and are associated with segregation in the vicinity of oscillation marks. These oscillation marks are regions in which high degrees of segregation of elements such as S, P and Mn can occur and have a role in the nucleation of transverse cracks and would also tend to favour the propagation of cracks. The grain size may often be coarse beneath the oscillation mark, and the notch like geometry will also tend to concentrate stresses.

High temperature ductility

Low ductility regions are believed to be the cause of cracking during continuous casting of steel. Crowther[31] has identified 4 distinct regions of low ductility as illustrated schematically in Figure 7. Those are

Region I/Type I - Embrittlement by Incipient Melting
Region IIa/Type IIa - Embrittlement by Second Phase Particles - (Mn,Fe)S
Region IIb/Type IIb- Embrittlement by Second Phase Particles - Nb(CN), AlN, V(CN)
Region III/Type III - Embrittlement by Transformation

Region I has been identified to occur at high temperatures, typically 20-50°C below the mean solidus temperature.[31] Fracture surfaces are characterised by inter-dendritic failure and the presence of particles such as MnS. The segregation of elements such as S to inter-dendritic regions during solidification resulted in incipient melting and formation of many types of defect in cast products including longitudinal surface cracking. In this region, the small subsurface cracks have been observed associated with oscillation marks.[26-32]

In the case of region II, the approximate temperature range is 1200-900°C depending on composition and test conditions.[31] The fracture propagates along austenite grain boundaries, and fracture surfaces show sometimes ductile dimples around the second phase particles in low ductility regions. These second phase precipitates are considered as (Mn,Fe)S for region IIa while Nb(CN), V(CN), Ti(CN) and AlN are associated in region IIb (Figure.7a). The high temperature end of this ductility trough is believed to be associated with the onset of recrystallisation. Nb delay recrystallisation more effectively than V either in solution or as precipitate, and this retardation of recrystallisation is believed to be responsible for extending the Type IIb ductility trough to higher temperatures. While type IIa low ductility is only apparent at quite high strain rates; the ductility is good at lower strain rates, or when there is an extended hold prior to testing.[33-35] In contrary to this, as shown in Figure 8[36] type IIb ductility is quite low as strain rate decreases. Type IIa ductility loss is dependent on composition, particularly Mn/S ratio and also is due to the precipitation of liquid FeS particles, and reduction of grain boundary decohesion due to S segregation.[33]

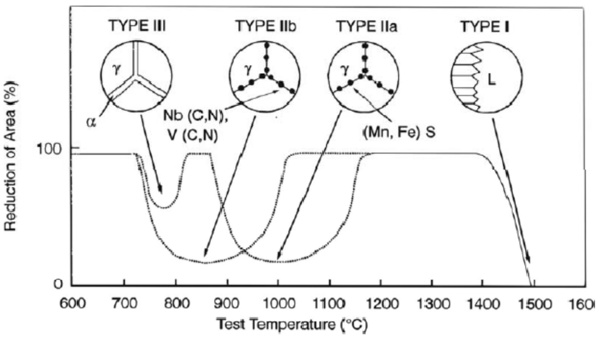


Figure 7. Schematic representation of temperature zones of reduced hot ductility of steel related to embrittling mechanism.[31]

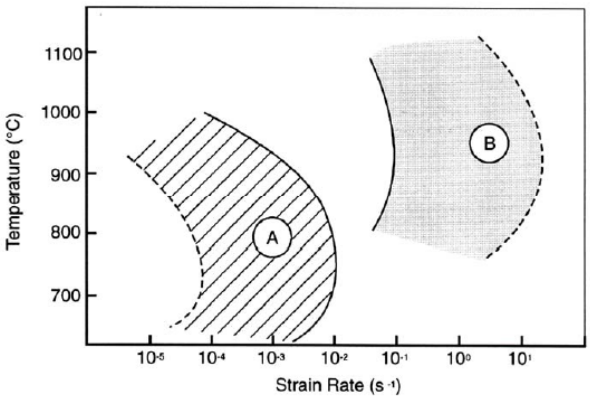


Figure 8. Regions of low ductility due to (a) precipitation of carbides/nitrides and (b) sulphides[36]

The composition of steel also influences transverse cracking. The Nb additions of as low as 0.01% has been reported to increase sharply transverse cracking in continuously cast slab, the propensity increases with increasing Al content.[32-37-38] Increased N and S also promote transverse cracking in Nb containing steels while C contents within the range 0.10- 0.17% are particularly prone to transverse cracking.[39] While additions of 0.2-0.3% Cu and Ni to the Nb containing steel have been also been reported to promote transverse cracking, additions of 0.02-0.04% Ti is required to reduce transverse cracks.

The last one is region III, which occurs over the approximate temperature range 900-600°C, depending on composition. If Type II low ductility is also present, then both of these two ductility troughs can merge together.[31] This region of low ductility is associated with the austenite to ferrite transformation and the associated fracture mechanism can be explained using the schematic shown in Figure 9.[40] On cooling below the transformation temperature, ferrite formation commences at austenite grain boundaries, leading to the formation of films of ferrite around the austenite grains. At temperatures within the transformation range, ferrite is softer, i.e., more ductile and has less strength than that of austenite, which is partly due to the higher atomic diffusivity of ferrite and to larger slip system of bcc (48) compared with fcc atomic structure (12).[41] Thus, when deformation commences, strain is concentrated at the primary ferrite film leading to weakening of grain boundaries, and the processes of ductile failure, i.e. void nucleation at second phase particles, and the growth of these voids, continues within the ferrite film.[42-44] Thus on a microscopic scale, fracture can be described as ductile, but overall the failure is brittle as fracture surfaces are characterised by intergranular failures although the facets of the individual grains are often associated with void formation around second phase particles. The high temperature end of the ductility trough is associated with the start of transformation, and is thus determined by composition and processing conditions. There appears to be a good relationship between the temperature at ductility starts to fall and the Ar3 temperature, the transformation temperature measured during cooling. It has also been suggested that the temperature at which ductility starts to fall is very close to the equilibrium transformation temperature Ae3, rather than the Ar3, as the deformation process accelerates the transformation kinetics.[45]

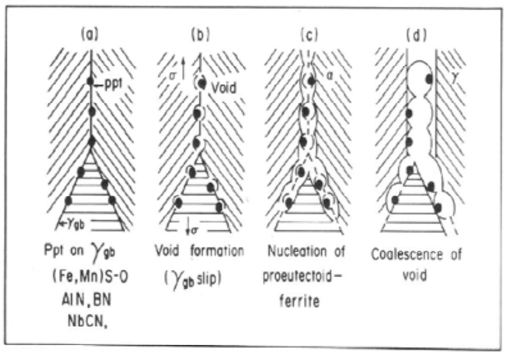


Figure 9. Mechanism for embrittlement in the low-temperature or two-phase zone.(40)

Ductility recovers at lower temperatures because the volume fraction of ferrite is higher, and the strain distribution between austenite and ferrite becomes more uniform. At lower temperatures, the strength differential between austenite and ferrite is also less, which will again contribute to a more uniform distribution of strain between austenite and ferrite. For ductility to recover completely, it appears that approximately 50% of the austenite must have transformed to ferrite.

Hot tearing

The strain to fracture of steel is less than 1% just below the solidus temperature i.e., in zone. A in Figure 7(b).[25] In this zone, the ductility in the interdendritic regions is reduced locally due to the microsegregation of S and P residuals at solidifying dendrite interfaces which lowers the solidus temperature.[25] The ductility remains effectively zero until the interdendritic liquids begin to freeze and thus called as 'zero ductility temperature' (ZDT), which lies within 30-70ºC of the solidus temperature as shown in Figure 10. This zone is responsible for hot tearing of steel. Application of any strain to the steel in this temperature region will propagate cracks outward from the solidification front between dendrites. The appearance of the fracture surface of resulted at this temperature zone exhibits a smooth, rounded facet, which is characteristic of the presence of a liquid film at the time of failure.[25] During transition from brittle to ductile behaviour, 100% ductility may not occur until some lower temperature approaches. As reported by Suzuki et. al,.[46] some embrittlement may be encountered at temperature as low as 1200ºC and the ductility is worsened due to the increased content of S, P, Sn, Cu and Si.

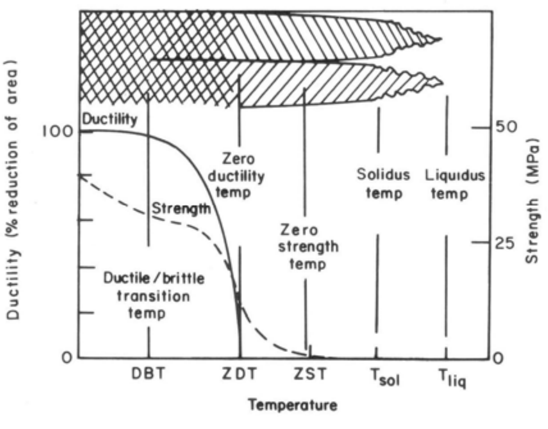


Figure 10. Relationship between mechanical properties in the high temperature zone of reduced ductility and corresponding schematic presentation of solid/liquid interface during casting.[25]

Techniques For Assessing Hot Ductility

Hot ductility is most commonly assessed using an elevated temperature tensile test. As such there is no standardized test available for assessing hot ductility. In view of this, different researchers have adopted different procedures for evaluating hot ductility. However, typically used thermal cycle involves heating the sample to a solution temperature in the range 1200-1350°C to produce a coarse austenite grain size and dissolve any microalloy precipitates like Nb(CN), V(CN), Ti(CN) etc., followed by cooling to a test temperature at a rate to simulate that experienced at the surface of a continually cast product. Typically cooling rate is kept 60°C/min although different researchers have kept different cooling rates. Results of some researchers show that increasing cooling rate to test temperature can reduce hot ductility. The strain rate of 10-3 to 10-4 s-1 is employed for straining to failure to simulate that experienced during the straightening of continuously cast slab. In micro alloyed steels, hot ductility is reduced on reducing strain rates.[43] However, as reported by Suzuki et.al,[39] in the region II, the precipitation of finely distributed oxy-sulfides at the austenite grain boundary weakens the boundary strength, and thus over-aging treatments such as slow cooling, holding for certain time, or slow rate of straining result in good ductility. In contrast to this, the embrittlement in the region III is manifested by the slower strain rate of test.[57] The solution temperature may affect the austenite grain size, and it is known that coarser austenite grain sizes will reduce hot ductility.[47]

For simulating the continuously cast condition more closely, some tests melt the test piece insitu prior to testing.[48-50] This is performed especially, when evaluating the effects of Ti and S on hot ductility as complete dissolution will not be achieved by a solid state heat treatment. Instead of continuous cooling to a test temperature, some researchers have employed more complex thermal cycles to simulate more accurately the complex temperature patterns experienced at the surface of a continuously cast product.[51-52] Banks et al.[53] have considered a more accurate simulation of the commercial cooling cycle involving both a rapid primary cooling followed by slower secondary cooling with and without temperature oscillations (Figure 11). Cooling cycles employing temperature oscillations have been shown to have a marked effect on hot ductility in some situations, by promoting precipitation of AlN.

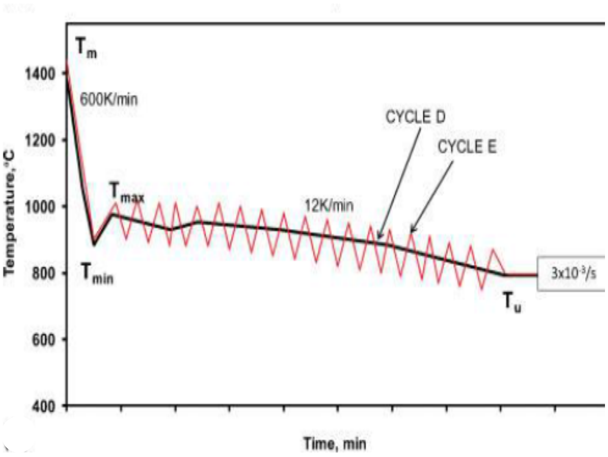


Figure 11. Schematic of Thermal cycles simulating continuous casting of slabs with and without oscillation.

Some tests have been also carried out in air after soaking for reproducing hot shortness effects due to copper.(54) Ductility is also assessed visually by examining any cracks on the surface with oscillation marks, which are known to be crack initiation sites for transverse cracks.

Relevance of Hot Ductility Test to Transverse Cracking

Some researchers have reported quantitative relationships between measures of hot ductility and transverse cracking. Bannenebrg(37) established a relationship between the number of transverse cracks per slab and the reduction of area in a hot tensile test. According to the findings of Bannenebrg,(37) no cracked slabs were observed above a reduction of area value of 75% while Suzuki et. al.,(55) have suggested a value of 60% reduction of area to avoid slab cracking. On the other hand, based on the finding of Mintz and Yue, (56) a reduction of area value of 30-40% is more realistic to avoid transverse cracking. Therefore, there is considerable discrepancy between the suggested values, and it is likely that such a value can only be ascribed to specific tensile test conditions and slab assessment methods. As well as the depth of the ductility trough and the temperature at which it occurs are also significant. If slab straightening can be carried out outside the temperature range of low ductility, then transverse cracking may be avoided.

Conclusions

Liquid embrittlement and hot shortness effect of Cu in Cu bearing steel has been reviewed.

- It involves penetration of liquefied Cu enriched phase at the steel/scale interface into austenite grain boundaries.

- This is accelerated under stress during hot rolling or forging at temperature higher than that of Cu enriched phase.

- The control of the microstructure near steel/scale interface by alloying the steel with Ni/Si will lead to reduce the amount of liquid Cu enriched phase.

- The technique employed by researchers for evaluating hot shortness effect is mainly hot tensile and hot bend test after oxidation.

- Cu bearing steels also exhibit transverse cracking in continuously cast products.

- Hot tensile test employing with a strain rate nearly similar to that of straightening operation during continuous casting of thick slabs has been also used for evaluating hot ductility behaviour and surface cracks of continuously cast products.

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