

## SYNOPSIS

Austenitic stainless steels find wide applications because of their good workability, weldability, corrosion resistance, and superior mechanical properties at elevated temperature. One of the important criteria used for the selection of these steels for specific applications is the type of phase changes occurring in them. In general, when exposed to elevated temperature, carbides followed by intermetallic phases may form in these steels, the type and the extent of precipitation at a temperature being sensitive to the composition. An important aspect of the precipitation of the intermetallic phases, particularly that of deleterious sigma, is the uncertainty in predicting their occurrence. The present work has been mainly devoted to an investigation of the phase instability in two grades of austenitic stainless steels and to assess, on this basis, their suitability for high temperature applications.

The steels studied are types AISI 316L and DIN 4981 (corresponding to AISI 347 but with about 2 pct. Mo and higher Ni contents) which in the present work have been referred to as Steel A and Steel B respectively. The phase instabilities have been studied in the quenched and aged

specimens - aging being carried out at 700° and 800°C upto 4000 hrs. The effect of cold work has also been examined using quenched specimens rolled to yield 20, 40 and 60 per cent deformation at room temperature before subjecting them to aging. The techniques used for this study are transmission electron microscopy, scanning electron microscopy, energy dispersion X-ray spectrometry and X-ray diffraction.

In Steel A at both the aging temperatures,  $M_{23}C_6$  has been observed to precipitate out followed by Laves, chi and sigma phases; in Steel B, on the other hand, no precipitation apart from  $Fe_3C$  and  $M_{23}C_6$  has been detected upto 4000 hrs. of aging and even with 40 per cent deformation. Both the precipitated carbides,  $M_{23}C_6$  and  $Fe_3C$  have the cube orientation relationship with the austenite matrix. The  $Fe_3C$  particles are very fine and appear to be spherical in shape.  $M_{23}C_6$  particles, however, assume specific non-spherical geometric shapes. Depending upon the precipitating sites which may be grain boundaries, incoherent and coherent twin boundaries, close to the incoherent twin boundaries or inside the grains away from twin boundaries, the particles have been observed to assume different morphologies. It has been observed that whatever be the morphology such as thin plate, lath,

polyhedron, cuboid, rhombohedron, etc. the  $M_{23}C_6$  particle/matrix interfaces always consist of  $\{111\}$  and  $\{110\}$  types of planes which have been determined by trace analysis. The nucleation of  $M_{23}C_6$  precipitated at different sites is discussed with the help of the existing models. The growth of these carbide particles leading to different morphologies are rationalised in the light of the ledge mechanism.

$M_{23}C_6$  precipitated at grain boundaries maintains orientation relationship with one of the grains constituting the boundary and on longer aging it grows gradually getting embedded into this grain and keeping one of its edges attached to the boundary. The growth of these particles is associated with the migration of austenite grain boundaries. Successive layers of  $M_{23}C_6$  particles formed close to the incoherent twin boundaries on both sides of the interface and the particles precipitated on coherent twin boundaries as thin plates have been analysed to have their broad faces on the twinning plane. The same feature is also displayed by the occasionally formed thin plates inside the austenite grains. Such a plate, however, has never been observed in the vicinity of an austenite grain boundary. Thin plates of  $M_{23}C_6$  have also been observed to form around undissolved NbC particles in Steel B, and a notable feature is that the broad faces of these plates



are always  $\{110\}$  planes and the bounding edges consist of  $\{111\}$  planes. From a single undissolved NbC particles more than one plates have been observed to form at times. The existing models about the lamellar plate formation in close proximity with the incoherent twin boundaries do not appear to explain all the specific features observed in this study, and it is suggested that the residual stress produced by quenching around twin boundaries and around undissolved NbC particles exerts influence on the formation of plates.

In Steel B, niobium carbide (NbC) has been observed to precipitate during aging, both in intergranular and intragranular manner. Inside the austenite grains NbC precipitates on dislocations, associated with stacking faults, and as dot-like particles. The observed features of these particles are broadly in agreement with those reported in the literature and are discussed making use of existing models. It has been noted that the lattice parameter of the precipitated NbC is less than that of undissolved NbC particles and this might be owing to the fact that precipitated NbC particles are of nonstoichiometric composition. The amount of dot-like precipitates of NbC in this steel appears to be higher compared to those reported in the literature and this is probably the effect of small addition of boron to this steel.

An interesting feature of precipitation in Steel B is that NbC particles which remain undissolved during solution treatment, gradually go into solution on longer aging while  $M_{23}C_6$  plates formed around these grow. At the same time precipitation of NbC in a fine form continues elsewhere. It is suggested that the urge for formation of Cottrell atmosphere and precipitation on the dislocations (either partial or undissociated), generated freshly around the precipitated particles becomes high making the matrix depleted in solute content and the larger undissolved NbC particles have to dissolve in order to supply the solutes.

Intermetallic phases such as Laves, chi and sigma have been observed to form in Steel A while none of them has been detected in Steel B on aging upto 4000 hrs., even in the cold worked specimens. Very often clusters comprising  $M_{23}C_6$ ,  $\chi$ , and  $\sigma$  have been observed on aging both in deformed and undeformed specimens. Cold work enhances the formation of chi and sigma phases; its influence on the formation of Laves phase, however, appears to be less. On longer aging, as the amounts of chi and sigma phases increase,  $M_{23}C_6$  gradually goes into solution. Evidence has also been obtained for the transformation of  $M_{23}C_6$  "in situ" to either chi or sigma phase.

On the basis of an analysis conducted on a large number of steels already investigated and reported in the literature, it is suggested that not only the "effective equivalent Cr content" but also the "relative amounts of chromium and other sigma promoting elements" in the matrix after the precipitation of carbides are important in assessing the sigma forming tendency of an alloy. The selection by the system, for precipitation of one or a combination of phases from out of laves, chi and sigma is found to be influenced by the type, apart from the "effective Cr equivalent", of alloying elements present.

The recrystallization behaviour of these steels has also been studied. Recrystallization of Steel B has been very much delayed compared to that of Steel A; with 40 percent deformation, recrystallization is completed after 500 hrs. of aging at 800°C in Steel A whereas in Steel B, even on aging upto 4000 hrs., it is not completed. Nucleation and growth of the recrystallized grains have been discussed in the light of existing models, and a mechanism is put forward for the nucleation in Steel B.

On the basis of the present results on phase instability and the reported mechanical and physical properties of the alloys of similar compositions in the literature, it may be concluded that the Steel B is a superior alloy for high temperature applications, compared to Steel A.