

# Precipitation and Hot Deformation Behavior of Austenitic Heat-resistant Steels: a Review

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**ABSTRACT:** Due to their favorable combination of high strength, corrosion resistance, perfect mechanical properties, workability, and low cost, austenitic heat resistant steels have been considered as important candidate materials for advanced supercritical boilers, nuclear reactors, super heaters, and chemical reactors. Because the precipitation behavior of steels over long-term operation at high temperatures may cause mechanical characteristics to deteriorate, it's critical to understand how secondary phases evolve in the microstructure of steels. An overview of recent advances in the precipitation behavior and coarsening process of different precipitates in austenitic steels during aging is presented here. MX carbonitrides, M<sub>23</sub>C<sub>6</sub> carbides, Z phase, sigma phase, and Laves phase are some of the secondary phases that occur under service circumstances. The coarsening rate of M<sub>23</sub>C<sub>6</sub> carbides is found to be much greater than that of MX carbonitrides. A constitutive equation may be constructed to explain the thermal deformation mechanism, and the resulting processing maps can be used to optimize thermal processing settings, resulting in better thermal processing characteristics of steels.

**KEYWORDS:** Austenitic Steels, Coarsening Behavior, Hot Deformation, Heat-Resistant Microstructure.

## 1. INTRODUCTION

Austenitic heat-resistant steels have been extensively utilized as structural materials for high-temperature applications such as boilers, nuclear reactors, super-heater tubes, and re-heater tubes in Ultra-Supercritical power plants since the early 1890s. Environmental protection and clean energy standards have prompted the use of ultra-supercritical (USC) facilities in recent years. Materials with excellent high-temperature capabilities should be developed because to the increased steam pressures and temperatures in USC equipment. Austenitic heat-resistant steels are gaining in popularity because to their better long-term high temperature strength, increased creep resistance, outstanding fabric ability, great resistance to oxidation and corrosion, and cheap cost.

Stainless steels are, in fact, the foundation of austenitic heat-resistant steels. Steels are mostly composed of iron, and chromium must be added to prevent rusting and corrosion in service conditions. Cr is added to increase corrosion and oxidation resistance as well as hardenability. In addition to Cr, stainless steels include a wide number of additional alloying elements, the presence of which improves certain characteristics. There is no question that the addition of Ni can improve austenite's stability. When significant quantities of Ni and Cr are added, the resulting steel is known as austenitic heat-resistant steel. The most apparent distinction between austenitic heat-resistant steels and stainless steels is that the former is often utilized at temperatures over 600 degrees Celsius [1]–[3].

The majority of austenitic heat-resistant steels fall into one of two categories: Types include 18Cr-8Ni and 25Cr-20Ni. Composition optimization has been explored in a number of contemporary austenitic heat-resistant steels. Type 347 and 321 steels were created by adding Ti, V, and Nb to basic Fe-Cr-Ni steels. Mo is added to increase pitting resistance. Cu and V are thought to contribute to the strengthening of austenitic steels in their own right. The strength of austenitic steels like Super304H and HR3C has been significantly improved by adding elements B, V, Mo, W, and Cu. By substituting N for Ni, novel austenitic heat-resistant steels as NF709 and SAVE25 have been created.

Austenite adorned with precipitates such as MX carbonitrides and M<sub>23</sub>C<sub>6</sub> carbides characterizes the microstructures of austenitic heat-resistant steels. The coarsening rate of M<sub>23</sub>C<sub>6</sub> carbides will be increased at high temperatures, reducing creep strength. It is obvious that changes in mechanical characteristics would result from precipitation and coarsening behavior of precipitates over long-term operation at high temperatures. As a result, the development and coarsening characteristics of later phases must be clarified.

Austenitic steels are now manufactured via thermal-mechanical techniques such as hot rolling, forging, and extrusion. Hot forging processes are used to create the majority of metallic materials. As a result, heat

deformation behavior will be a significant factor in the manufacturing process. Since its introduction by Prasad et al., the processing map has evolved into a useful tool for analyzing workability and optimizing processing parameters in hot working. As a result, a lot of research is being done on the hot deformation behavior of austenitic steels using processing map technology [4]–[6].

As previously stated, composition changes are always beneficial to the development of austenitic heat-resistant steels. The microstructure development in austenitic steels is receiving more interest these days. The precipitation and coarsening behavior of precipitates in austenitic heat-resistant steels are discussed in this article. The hot deformation processes of austenitic heat-resistant steels will also be addressed, as they play an important role in improving workability and microstructure control.

## 2. PRECIPITATION BEHAVIORS OF SECOND PHASES DURING AGING

Precipitation hardening, solution hardening, and dispersion hardening may all be used to strengthen austenitic heat-resistant steels. Fine nanoparticle precipitates in a metal matrix would provide the materials a high creep strength. At temperatures about 700 °C, isothermal aging may increase the precipitation of secondary phases, which typically lead to changes in mechanical characteristics. As a result, secondary phase development must be identified and comprehended. The microstructure is characterized by refined MX carbonitrides distributed in an austenitic matrix, and large densities of twins appear at random during aging.

A small amount of M<sub>23</sub>C<sub>6</sub> phase may be found in certain steels, particularly those with a high chromium concentration. Within austenitic heat-resistant steels, Z-phase, sigma phase, and laves phase may develop over time. Many studies have attempted to model precipitation kinetics in austenitic heat-resistant steels, in addition to using experimental methods. The percentages of equilibrium phase in 15Cr-15Ni austenitic steel were calculated using the MatCalc thermo-kinetic software program. MX carbonitrides, M<sub>23</sub>C<sub>6</sub> carbides, Z phase, Cr<sub>2</sub>N, and Cu precipitates are the most common precipitate phases in austenitic steels (M in MX denotes Nb, Cr, Mo or Fe; M in M<sub>23</sub>C<sub>6</sub> stands for Cr). M<sub>7</sub>C<sub>3</sub> carbides may be found in Fe-Cr-C alloys as well [7]–[9].

### 2.1. In austenitic steel, secondary phases include:

#### 2.1.1. MX carbonitrides, version:

In general, nano-sized block-shaped MX carbonitrides are nucleated densely along dislocations, which grow in size as the isothermal holding duration increases. Short-term annealing at temperatures about 700 °C was used to study the production of MX carbonitrides, and MX carbonitrides were found as intragranular carbonitrides with fcc crystal structure. The precipitation of these carbonitrides would cause dislocation motions to be slowed, which would enhance creep strength at high temperatures. Ti, Nb, and V have a greater affinity for carbon than chromium, which is advantageous for the production of MX carbonitride. To prevent the development of M<sub>23</sub>C<sub>6</sub> carbides, strong carbide-forming alloying elements are typically added. Furthermore, the increased nitrogen concentration may lead to the production of MX carbonitride. MX carbonitrides, in particular, are the most common strengthening precipitates in austenitic heat-resistant steels.

#### 2.1.2. M<sub>23</sub>C<sub>6</sub> phase:

M<sub>23</sub>C<sub>6</sub> carbide with fcc crystal structure is another common deposit in austenitic steels. Several recent studies have looked at the connection between M<sub>23</sub>C<sub>6</sub> and grain boundary serration. After long-term exposures at temperatures about 700 °C, rod-like M<sub>23</sub>C<sub>6</sub> carbides have been frequently seen near grain boundaries, and M<sub>23</sub>C<sub>6</sub> precipitation may induce intergranular corrosion susceptibility. M<sub>23</sub>C<sub>6</sub> carbides nucleate and develop along grain boundaries in most cases. M<sub>23</sub>C<sub>6</sub> phases may also be seen in regions with a high density of dislocations. Rod-shaped M<sub>23</sub>C<sub>6</sub> distribution along dislocation lines in S31042 austenitic heat-resistant steel. The discontinuous M<sub>23</sub>C<sub>6</sub> particles with tiny sizes at grain borders, on the other hand, may contribute to grain boundary sliding resistance, resulting in increased creep strength.

#### 2.1.3. NbCrN phase (Z phase):

Z phase is a small-sized precipitate that forms in Nb- and N-containing austenitic heat-resistant steels. The following are the orientation relationships between the Z phase and the austenite matrix: [010]<sub>z</sub>/[110], [111]<sub>z</sub>/[100], and [001]<sub>z</sub>/[001]. Long-term exposure to high temperatures causes a significant quantity of Z

phases to form within the grains of S31042 austenitic steel. The granular and triangular Z phases were detected at grain and twin boundaries in TP347H austenitic heat-resistant steel when the holding duration was extended to 2200 h at 700 °C, with the size of the Z phase ranging from 100 to 200 nm. According to certain studies, the development of the Z phase is linked to matrix precipitation or transition from Nb-containing MX phases. Due to its great stability, Z phase has been shown to strengthen S31042 steel in certain studies. In reality, when the Z phase is refined and evenly distributed in the matrix, it may reinforce the matrix and delay recrystallization. Coarse Z phase, on the other hand, may impair steel fatigue characteristics [10].

#### 2.1.4. Phase of Sigma:

The brittle sigma phase has a tetragonal crystal shape and is a typical intermetallic phase. The TEM micrograph of the sigma phase in austenitic steel after 240 hours of aging at 750°C. The orientation connection between the sigma phase and the austenitic matrix is not clear, although it is often characterized as (111)/(001) and/. When the sigma phase precipitates on the grain boundaries, the creep characteristics decrease. The presence of the sigma phase in austenitic heat-resistant steels reduces both the toughness and the corrosion resistance of the steels. The elements iron, chromium, molybdenum, and niobium may aid in the creation of the sigma phase. In general, there are three methods to get the sigma phase to precipitate in austenitic heat-resistant steels:

- (1) austenite transformation,
- (2) ferrite transition,
- (3) M<sub>23</sub>C<sub>6</sub> carbides are transformed.

Chromium was enhanced in the austenite zone close to the grain boundaries. The ferrite phase may develop at a later stage of aging, resulting in the precipitation of the sigma phase near grain boundaries.

#### 2.1.5. Phase of the Laves:

When Nb-containing austenitic heat-resistant steels are subjected to temperatures between 700 and 900 degrees Celsius, the Laves phase (Fe<sub>2</sub>Ti and Fe<sub>2</sub>Nb) forms. The boron addition may lead to the development of Laves phase, which typically precipitates near grain boundaries. Laves phase embrittlement occurs when the Laves phase precipitates, resulting in a loss of toughness, ductility, and corrosion resistance. Laves phase at grain boundaries in Fe-20Cr-30Ni-2Nb steels TEM micrograph. Yamamoto et al. proposed that the Fe<sub>2</sub>Nb Laves phase could serve as a strengthener to enhance creep resistance, based on the high thermal stability of Fe<sub>2</sub>Nb in austenitic matrix.

The dynamic precipitation of Fe<sub>2</sub>Nb in the creep course would effectively pin dislocations, thus improving creep resistance. The size of the Fe<sub>2</sub>Nb phase can be refined by adding Si, however there was no evidence of improved creep performance compared to the alloy without Si. Because a single Fe<sub>2</sub>Nb Laves phase cannot provide a satisfactory strengthening effect, alloys containing MC-type carbides and intermetallic Fe<sub>2</sub>Nb showed better creep resistance. The refining of stable Fe<sub>2</sub>Nb particles to less than 100 nm should be given special attention. It should also be noted that not all austenitic heat-resistant steels may produce the Laves phase. Laves phase was not detected in TP347H austenitic steel after 2200 hours of aging at 750 °C, indicating that the development of Laves phase is dependent on the steel composition and service circumstances.

Using just optical microscopy (OM) or SEM micrographs, it is difficult to determine what kind of precipitate phase will develop in the matrix. The use of TEM, EDS, and X-ray diffraction (XRD) analytical techniques to identify fine secondary phases such as MX carbonitrides, sigma phase, Laves phase, and Z phase, among others, is critical. The quantity of precipitates, their size, shape, and distribution in the matrix all have an impact on the mechanical characteristics of austenitic heat-resistant steels. In austenitic heat-resistant steels, homogeneous dispersion of small-sized precipitates may play a role in precipitation strengthening. Large-sized phases near grain boundaries, on the other hand, are detrimental to the steel's characteristics.

### 3. DISCUSSION

Secondary stages, without a doubt, may help with creep resistance. Long-term service at relatively high temperatures, on the other hand, often causes coarsening of these phases, resulting in a loss of strength in austenitic heat-resistant steels. All degradation processes in steels, such as recovery and recrystallization, grain and sub-grain development, and coarsening of precipitates, are known to be accelerated by a rise in service temperature. Steels' mechanical characteristics are influenced by the size, volume fraction, shape, and distribution of precipitates. Fine particles with long-term durability at high temperatures should be evenly dispersed throughout the matrix to enhance creep characteristics. As a result, the coarsening behaviors of MX carbonitrides and the M<sub>23</sub>C<sub>6</sub> phase were thoroughly studied. Characterizing and simulating the nucleation, growth, and coarsening of M<sub>23</sub>C<sub>6</sub> phase and MX carbonitrides in austenitic heat-resistant steels has been a goal of past study.

Many studies of austenitic heat-resistant steels have concentrated on creep behavior and microstructure development at high temperatures in recent decades, but little attention has been given to the steels' thermal deformation performance and workability. In reality, hot deformation processing maps are advantageous for improving workability and controlling material microstructures. The hot working properties of 321 steel and 304 steel were studied using constitutive equations and processing maps. In austenitic steels, heat deformation resulted in smaller grains with thinner carbide coatings around them. The martensitic changes may be induced by deformation in high purity Fe-Cr-Ni austenitic steels. The reduced peak flow stress in 316LN austenitic heat-resistant steel is due to the greater deformation temperature and lower strain rate.

The constitutive equation, which includes flow stress, strain rate, strain, and temperature, may be used to explain the plastic flow characteristics of steels and forecast high-temperature flow behaviors under experimental deformation circumstances. Furthermore, an exponent-type Zener-Hollomon equation may be used to describe the effects of temperature and strain rate on hot deformation behavior. The microstructure of S31042 austenitic heat-resistant steel experienced deformation instability, dynamic recovery, partial dynamic recrystallization, full dynamic recrystallization with equiaxial structure, and final dynamic recrystallization with mixed crystal structure as the Zener-Hollomon parameter decreased.

The gray unstable zones can be observed to be mostly located in areas where the strain rate is quite rapid and the deformation temperatures are relatively low. The unstable zone and safe zone of the materials may be graphically shown using a thermal processing map. Dynamic recrystallization regions, superplasticity areas, and dynamic recovery areas are all examples of safe zones. During hot working, unstable zones should be avoided, and flaws such empty zones, wedge zones, grain boundary cracking zones, and adiabatic shear band formation regions are bad for the steel's properties.

Furthermore, the safe zone of TP347H austenitic steel is mostly made up of deformation circumstances including a deformation temperature of about 1000 °C and a strain rate of 1 s<sup>-1</sup> or less. The processing maps of austenitic heat-resistant steels have been described in many studies. The optimum hot-working state for 316LN in the range of experimental deformation parameters occurs at a temperature of 1200 °C and a strain rate of 0.001 s<sup>-1</sup>, according to the power dissipation map of 316LN. The optimal hot working regimes for Fe-18Mn-18Cr-N austenitic steel were determined to be in the range of 1050 °C to 1100 °C, with a strain rate of 0.01 s<sup>-1</sup>. They also addressed the causes of intergranular cracking in terms of Cr<sub>2</sub>N particle precipitation between granules.

Furthermore, during thermal deformation, the deformation temperature and strain rate are two important factors that influence the microstructure development of austenitic steels. According to the findings, increasing temperature promotes dynamic recrystallization, and the quicker the strain rate, the smaller the grain size would be. The impact of thermal deformation on the precipitation behavior of MX carbonitrides have received little consideration. In comparison to samples without thermal deformation, more MX carbonitrides will precipitate in the austenite matrix, and steels deformed in stable areas will have more precipitates than steels deformed in unstable sections.

#### 4. CONCLUSION

Microstructure development and hot workability of austenitic heat-resistant steels are strongly related to their characteristics under service circumstances. The precipitates in austenitic steels during aging at high temperatures are addressed in this article, showing the precipitation hardening process. MX carbonitrides, M<sub>23</sub>C<sub>6</sub> carbides, Z phase, sigma phase, and Laves phase are all investigated in the second phase. In fact, identifying the kind of phase using SEM micrographs is difficult, and additional test techniques are required. MX carbonitrides are helpful to increasing the strength of steels because of their thermal stability. While it is difficult to say if phases such as M<sub>23</sub>C<sub>6</sub>, sigma, Z, and laves phases are beneficial to steels. M<sub>23</sub>C<sub>6</sub> carbides are detrimental phases in austenitic steels because they are simple to nucleate and develop quickly along grain boundaries. The Sigma and Laves phases are fragile and have a negative impact on performance. During service, the bulk of the harmful phases tend to coarsen. Controlling the size, distribution, and form of the secondary phases is the greatest method to enhance the characteristics of austenitic heat-resistant steels. The behavior of austenitic steels during heat deformation is examined. The optimal process parameters may be readily found using the processing maps. The microstructure and performance of steels may be further customized by using appropriate heat deformation.

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