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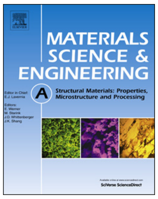

# Microstructural bandings evolution behavior and their effects on microstructure and mechanical property of super-austenitic stainless steel

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## ABSTRACT

Bands were cut from surface and center sections (denoted as SS and CS, respectively) from a super-austenitic stainless steel cast slab, and were hot rolled to be 3 mm thick plates under the same conditions. It was found that the SS hot-rolled plate possesses different microstructural bandings (MSBs) from those in the CS plate. Formation mechanisms of MSBs and precipitates during hot rolling and their evolution behavior during solution treatments (1000–1150 ℃ for 30 min) were investigated. The results show that the SS plate has slight MSBs with no precipitates, while the CS plate has high density of MSBs consisted of massive σ phases. The MSBs in SS and CS plates were found to be vanished after solution treatments at 1000 ℃ and 1100 ℃, respectively. Also, length fractions of low-ΣCSL boundaries of Σ3, Σ9 and Σ27 in both SS and CS plates were observed to increase and then decrease with increasing temperatures. The MSBs could have affected the evolution of grain boundaries, and changed the length ratio of CSL boundaries to the total grain boundaries. Numerous zonal structures composed of fine grains were observed in CS plates after solution treatment. Precipitates inside the MSBs could retard the boundaries movement during recrystallization to refine the grain. Tensile tests indicate that the strength of hot-rolled and solution-treated CS plates was higher than that of SS plates by 2–7%, and the elongation reduced by 2–5%. The main reasons were that the smaller average grain sizes and the precipitates in MSBs of CS plates enhanced the strength but the precipitates deteriorated the elongation.

## 1. Introduction

Super austenitic stainless steels (SASSs) are widely applied in many industrial fields, such as the chemical, petrochemical, oceanic, nuclear, military and other industries, due to their excellent combination of corrosion resistant, weldability and mechanical properties [1–3]. The traditional industrial production method for SASS is continuous casting, which possesses low solidification rate. As is well known, solute segregation is an inherent characteristic of alloy solidification. Because solidification proceeds from the casting surface to the center, the segregation behavior becomes more and more serious toward the center. Réger et al. [4] indicated that macro-segregation in castings is non-uniformity of composition over macroscopic or large areas with the size varies from a few hundred to several thousand microns. Such macro-segregation can be extended throughout the length of castings. Due to the large sizes, macro-segregation is considered more detrimental than micro-segregation to the ultimate properties of products, as it cannot be eliminated even with prolonged homogenization treatments [5]. For SASS, the high contents of Cr and Mo promote the rejection of these elements to the inter-dendritic regions (IDRs) during the solidification process [6,7], and thus facilitate the formation of eutectics or second-phases [7,8], especially in the center of castings. During subsequent rolling, these IDRs rich in solute elements become elongated and form chemical composition bands aligned parallel to the rolling direction. Upon cooling, these high alloying element content bands and the neighboring low alloying element bands together constitute microstructural bandings (MSBs) within the hot-rolled plates [9]. Similar phenomena are also found in previous literature [10–13]. In order to understand this phenomenon, much work on MSBs themselves and their effects on the properties have been done on steels and alloys in the past few decades. For instance, Stauffer et al. [9] examined the presence of MSBs and their effects on the tensile properties in AL-6XN SASS. Similarly, Adams et al. [14] studied the influence of centerline sigma on the through thickness toughness and tensile properties of AL-6XN SASS. Both of the results show that MSBs, which mainly consist of brittle sigma particles, remarkably deteriorate the mechanical properties in the short transverse direction. Fonda et al. [7] studied the 3D distribution, interconnectivity, morphology of coarse precipitates and the internal voids network associate with these precipitates by X-ray microtomography in a SASS. The results indicated that almost every coarse

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sigma particle is associated with at least one void, and the presence of sigma-plus-void has important implication for the mechanics of this alloy. This result may explain why SASS ingots are prone to cracking during hot rolling deformation. Moreover, Li et al. [15] investigated the effect of solution treatment on the microstructure evolution in the segregation area of S31254 SASS plate. The results shown that two different secondary particles, i.e. sigma phase and chi phase, precipitate and disperse in different positions in the matrix of the studied alloy when the solution temperature lower than 1150 °C. Also, many other relative studies of the segregation and the precipitate in SASS also can be found in previous literature [4,5,7,16]. However, comparative studies on the evolution of MSBs including and excluding precipitates, and their effects on the microstructures and mechanical properties during hot deformation as well as solution treatments have not been systematically reported for the studied SASS.

In this paper, bands were cut from surface and center sections (named as SS and CS, respectively) from the studied SASS cast slab, and were hot rolled under the same conditions by using layered rolling experiments to prepare SS and CS plates, from which MSBs in SS plate excluding precipitates while MSBs in CS plate including precipitates were obtained. Therefore, there are three clear goals according to the above analyses. The first is to identify the formation mechanisms of MSBs including and excluding precipitates from the cast slab to the hot-rolled plates. The second is to investigate the evolution of MSBs and their effects on the evolution of coincidence site lattice (CSL) boundaries and the distribution of grains during solution treatments. The third is to compare the mechanical properties of hot-rolled and solution-treated SS and CS plates in consideration of MSB, CSL and the average grain size (AGS)

## 2. Material and experimental procedures

The chemical compositions of the studied material (wt%) were 0.03 C, 0.023 P, 0.005 S, 0.215 N, 0.354Si, 0.684Cu, 0.452Mn, 22.4Ni, 19.4Cr, 6.65Mo and balance Fe, which were very similar to those of AL-6XN alloy (UNS N08367) [7,9,14]. The used material was continuous cast slab with a thickness of 150 mm. As shown by schematics in Fig. 1, section A, cut from the original cast slab, was used to study the solidification microstructures before hot deformation. To avoid hot cracking during hot rolling as mentioned above, section B, cut from the original cast slab, was hot forged into about 78 mm at 1250 °C. The forged slab was equally cut into three parts parallel to the casting surface with a thickness of 24–25 mm to prepare SS and CS slabs. After heating to 1200 °C and holding for 60 min, SS and CS slabs were hot rolled into ~3 mm-thick-plates under the same rolling procedures. The hot rolling schedule was 24–25 mm→17 mm→12 mm→8 mm→5 mm→3 mm. In order to prevent temperature loss too much during the deformation, the hot rolling process was carried out as soon as possible. After that, the hot-rolled plates were immediately water quenched to room temperature in 15 s after hot deformation to preserve the current microstructures and inhibit the formation of precipitates upon cooling processes. The initial and finishing rolling temperatures were about 1190 °C and 993 °C, respectively, which were measured by thermal infrared imager. It is noted that a contrastive specimen passed the same thermal cycle without deformation was adopted to see the formation of precipitates. SS and CS hot-rolled plates were then solution treated at 1000–1150 °C (interval: 50 °C) for 30 min, and then immediately water quenched to preserve the current microstructures and prohibit the formation of secondary phases during cooling process. These hot-rolled and solution-treated SS and CS plates were utilized to fabricate the specimens for microstructure, electron back-scattered diffraction (EBSD), transmission electron microscopy (TEM) analyses and tensile experiments.

A ZEISS ULTRA 55 scanning electron microscopy (SEM) equipped with an energy dispersive spectroscopy (EDS) was used to obtain microstructure and EBSD information at 20 kV with the work distance of 14.5 mm. It is noted that each result of the chemical composition in this work was the average value of multiple precipitates to ensure the accuracy of analyses. Specimens for microstructure observation were prepared using standard mechanical grinding and polishing, followed by electrolytic etching in a 60 pct $HNO_3$/40pct $H_2O$ solution at 6 V DC for 40–50 s. Specimens for EBSD analyses were electrochemical polished in a solution of 15.3% $HClO_4$ in ethyl alcohol at 23 V DC for 20–30 s after standard mechanical ground. In order to ensure the accuracy of EBSD results, the scanning area was as large as 1732.5 μm × 2310 μm and the step size was set at 0.3–3.5 μm depending on the grain size. The well-known Brandon criterion [17] was adopted to assess the distribution of CSL boundaries. Random high angle boundaries (HABs) and low angle boundaries (LABs) were defined as boundaries having misorientation angles $\geq 15^\circ$ and 5–15°, respectively. The fraction of each type of grain boundaries was determined based on the length fraction in all grain boundaries [18]. The value of AGS was measured by using the mean linear intercept method [19,20]. More detailed and definitive information about the microstructures and precipitates was performed using a FEI $G^2$ F20 TEM with an acceleration voltage of 200 kV. Specimens with diameter of 3 mm for TEM analysis were ground to ~50 μm and then electrolytically jet polished in a solution containing 12.5% $HClO_4$ and 87.5% ethyl alcohol at a temperature of $-20$ °C and a voltage of 32.0 V. Tensile tests were performed at room temperature with the strain rate of $1 \times 10^{-3}\ s^{-1}$ [21,22]. The dimensions of the tensile specimens were 50 mm in gauge length and 12.5 mm in parallel width. Each of the final value was determined by the average value of three identical heat-treated specimens.

## 3. Results and discussion

### 3.1. MSBs and precipitates formation mechanisms during hot deformation

### 3.1.1. Formation mechanism of MSBs

Fig. 2a and b show the as-cast structures of SS and CS slabs, respectively. Fine dendrites were the principal component for the SS slab, while coarse dendrites with massive eutectics in IDRs were the main components for the CS slab. The main reason for the aforementioned difference was the solidification rate during solidification process. It is well accepted that molten metal in the center solidified more slowly than that in the surface. More time was supplied for solute elements, i.e. Cr and Mo, segregate to IDRs and thus promote the formation of eutectics. Therefore, massive eutectics were detected in the CS slab. Eutectics in this alloy are the combination of austenite and intermetallic, which have been clearly established in literature [6]. The magnification of eutectics, as shown by subset in Fig. 2b, was similar to those reported in previous literature [6,23], and the chemical compositions of the intermetallic (point 1) were given in Table 1. After hot deformation, great

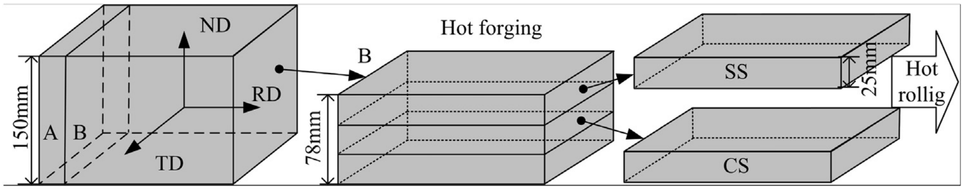

**Fig. 1.** Schematics of layered hot rolling experiments.