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Ultrafine grained microstructure tailoring in austenitic stainless steel for enhanced plasticity

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ABSTRACT

Using repeated or cyclic annealing process a recrystallised ultrafine grained microstructure possessing high strength and a reasonable ductility was obtained from a heavily cold rolled Austenitic Stainless Steel. Johnson Mehl Avrami and Kolmogorov model for predicting recrystallisation process was used to optimize the process parameters for the formation of ultra-fine grained recrystallised microstructure. The microstructure evolving during thermal cycling was analyzed by electron microscopy. The tensile properties of various ultrafine grained microstructures were determined. Electron microscopy studies on tensile deformed specimens showed that there is a narrow dispersion in grain size distribution of ultrafine grained microstructure which promotes shear banding or strain localization causing impairment of the ductility or poor plastic strain hardenability.

1. Introduction

In general, strengthening methodologies impair the ductility of metals and alloys. Many of the engineering materials are used in the polycrystalline state and, therefore, its properties are influenced by several microstructural parameters. Application of thermo-mechanical processes, a combination of heat treatment and deformation, are very effective in tailoring microstructure of steels. It was possible mainly owing to the phase transformation characteristics of the steels. However, many of the austenitic stainless steels (SS) grades do not exhibit phase transformation during annealing process. The absence of phase transformation in this class of SS renders them non conducive to microstructure engineering through thermo-mechanical process routes. In the absence of multi-phase microstructural constituents, austenitic SS's microstructure engineering is mostly limited to grain refinement and substructure strengthening through hot rolling in the no recrystallisation region [1,2]. Recent developments in thermo-mechanical processes suitable for austenitic SSs have led to the production of nano or ultrafine grained austenitic microstructure by a method of reverse transformation of strain-induced martensite (SIM) [3–14]. This technique involves martensitic transformation of metastable austenite by heavy cold rolling and then annealed to form recrystallised ultrafine austenite grains by phase reversion of martensite to austenite. The bulk ultrafine-grained (UFG) materials exhibit excellent tensile strength due to the Hall–Petch effect, wherein the advantage of grain boundary strengthening effect was taken [15,16]. Unfortunately, they suffer from low tensile ductility. The total elongation of UFG materials shows an improvement of up to 10%, better than nano structured (NC) materials [17]. However, when compared to their coarse grained state most of the NC and UFG materials show poor uniform tensile deformation [18,19]. Various strategies were suggested to overcome these obstacles with the aim to jointly improve ductility and strength [20–22]. One of these methodologies was to embed coarse grains in a NC/UFG matrix towards enhancing plastic deformation characteristics. This method was very successfully adopted in steels. It is assumed that in this type of microstructure modification, high strength is derived from the nano-grained matrix and coarse grains enhance the ductility. Hence, tailoring of the grain size distribution commonly referred to as bimodal or multimodal, in enhancing the ductility is increasingly finding its way in the development of UFG materials [23–27].

Our present research work was mainly devoted to tailoring of grain size distribution of an austenitic SS by the application of a thermo-mechanical process route for obtaining a good combination of strength and ductility. An important point to note about the commercial grade of AISI 304L austenitic SS of this research work was that it is not readily amenable to phase reversion process as reported by various authors [1–5]. The chemistry of this grade of SS allows only a limited martensitic transformation by cold

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deformation at room temperature. Therefore, we have adopted an alternative approach of thermo-mechanical process that was to utilize the stored energy induced by cold working for recrystallising ultrafine grain microstructure [28,29]. We have used very short repeated annealing cycles to inhibit/retard discontinuous recrystallisation that is normally observed in materials with low stacking fault energy during isothermal annealing [30]. The discontinuous recrystallisation process is not a favorable mechanism of grain refinement to submicron size because it promotes selective grain growth by annihilating smaller grains. Design of the process was based on the various observations made by the authors elsewhere [28,29]: (a) nucleation will preferentially occur in high strain energy regions during the first few cycles and set in an increased strain heterogeneity between recrystallised and residual deformed regions, and (b) on subsequent thermal cycles, nucleation takes place in the residual deformed region, as these are the more potential sites owing to their high stored strain energy. Therefore, under all probabilities the abnormal grain growth may be prevented depending on annealing temperature and time. In view of the above, a detailed study of the recrystallisation behavior of the cold rolled structure that is relevant to UFG formation was looked into. Further, we have made an attempt to establish the mechanisms of plastic deformation which would lead to an enhancement in ductility of a bulk UFG austenitic SS in bimodal type distribution.

2. Experimental details

2.1. Materials and processing

Commercial garde AISI 304L austenitic stainless steel plates with chemical composition as shown in Table 1 was solution treated at 1080 °C. Solution annealed plates were rolled at room temperature by multi-pass unidirectional rolling to a total of 90% reduction in thickness. 1 mm thick cold rolled sheets were annealed between the temperature range of 775–950 °C in an ambient furnace atmosphere. A short annealing durations (30–45 s, 60 s) were used before free cooling in the air by taking specimens out of the furnace. In this study, several repetitions of these short annealing cycles were tried to achieve the desired grain size or its distribution in the microstructure.

2.2. Materials characterization

Microstructure and phase transformation behavior of the specimen in as cold rolled state and after repeated annealing or thermal cycles was characterized Standard metallographic techniques were employed for the preparation of the specimens for optical microscopy. To reveal the microstructure, specimens were etched with a solution of 4 gms copper sulphate in 20 ml Conc. HCl and 20 ml water. The specimens for phase transformation studies were prepared by standard metallographic techniques and then electro-polished using a bath of acetic acid and 10% perchloric acid. Phase identification of electro-polished specimens was performed by using X-ray diffraction (XRD) technique using Bruker AXS D8 Discover diffractometer. Cu Kα of 0.154 nm wave length along with fast detector Lynx Eye was used for diffraction experiments. Transmission electron microscope (TEM) was used to examine the microstructure changes in annealed specimens. Thin foils from the specimens were prepared by initial careful mechanical thinning. Final thinning for electron beam transparency was achieved by electro-polishing using a bath of acetic acid and 10% perchloric acid. Microstructures were analyzed for grain size distribution by linear intercept technique.

Electron backscattered diffraction (EBSD) was performed with a JEOL 6500 F field emission gun scanning electron microscope (FEG-SEM). An EDAX/TSL EBSD system equipped with a high-speed Digiview camera was used for the diffraction pattern acquisition. All samples observed here were first prepared using the standard metallographic techniques and then polishing on Silica gel. Further, these were electro-polished in order to obtain the highest diffraction Kikuchi pattern quality. A step size of 100 nm was used for overview studies and 20 nm was used for high resolution EBSD.

2.3. Tensile property evaluation

The flat tensile dogbone-shaped specimen of 25 mm gauge length as per the ASTM: E-8M was machined from the cold rolled and the cyclic thermal processed specimens. Tensile tests were conducted at room temperature under displacement control at a strain rate of $1.3 \times 10^{-4}\,s^{-1}$ using Instron 8862 system of 100 KN capacities.

3. Results and discussion

3.1. Microstructure and structure of Cold rolled SS

The optical microstructure analysis of SS after cold rolling revealed, heavily deformed elongated grains in the rolling direction. The XRD phase analysis revealed the occurrence of partial SIM transformation of austenite by cold rolling. Volume fraction of the SIM was quantified by using Ferritscope and it was found to be 43% of volume fraction. Results of microstructure and SIM transformation analysis is shown in Fig. 1

3.2. Recrystallisation behavior of cold rolled structure during isothermal annealing

The grain refinement in this study was carried out by way of recrystallisation of a heavily deformed microstructure by repetitive annealing treatment. Hence, it was considered important to study the effect of isothermal annealing on the recrystallisation kinetics of cold deformed structure. Di Schino et al. [31] proposed a model

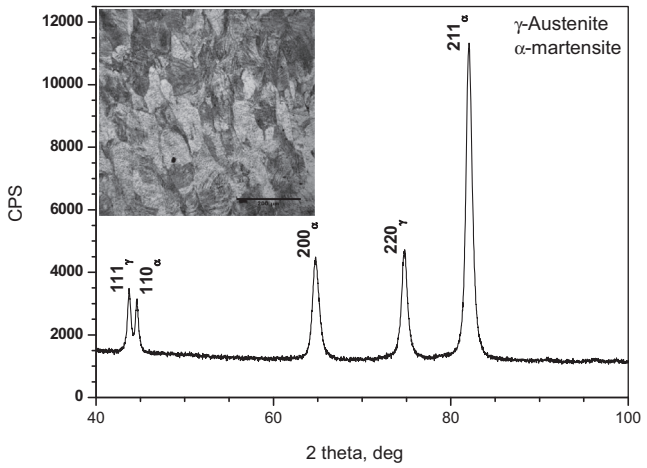

Fig. 1. X-ray diffractogram of the cold rolled specimen showing presence of strain-induced martensite in addition to parent austenite phase. In-set picture shows the microstructure of cold rolled specimen. Here rolling direction is along diagonal direction in micrograph.

Table 1
Chemical composition of the stainless steel.

Element	C	Mn	Cr	Ni	Si	S	P	N
wt%	0.02	1.5	18.6	10.1	0.3	0.01	0.028	0.02