Influence of Annealing Treatment on the Formation of Nano/Submicron Grain Size AISI 301 Austenitic Stainless Steels

D.L. JOHANNSSEN, A. KYROLAINEN, and PJ. FERREIRA

Nano/submicron austenitic stainless steels have attracted increasing attention over the past few years due to fine structural control for tailoring engineering properties. At the nano/submicron grain scales, grain boundary strengthening can be significant, while ductility remains attractive. To achieve a nano/submicron grain size, metastable austenitic stainless steels are heavily cold-worked, and annealed to convert the deformation-induced martensite formed during cold rolling into austenite. The amount of reverted austenite is a function of annealing temperature. In this work, an AISI 301 metastable austenitic stainless steel is 90 pct cold-rolled and subsequently annealed at temperatures varying from 600 °C to 900 °C for a dwelling time of 30 minutes. The effects of annealing on the microstructure, average austenite grain size, martensite-to-austenite ratio, and carbide formation are determined. Analysis of the as-cold-rolled microstructure reveals that a 90 pct cold reduction produces a combination of lath type and dislocation cell-type martensitic structure. For the annealed samples, the average austenite grain size increases from 0.28 μm at 600 °C to 5.85 μm at 900 °C. On the other hand, the amount of reverted austenite exhibits a maximum at 750 °C, where austenite grains with an average grain size of 1.7 μm compose approximately 95 pct of the microstructure. Annealing temperatures above 750 °C show an increase in the amount of martensite. Upon annealing, (Fe, Cr, Mo)23C6 carbides form within the grains and at the grain boundaries.

I. INTRODUCTION

AUSTENITIC stainless steels (SS) are frequently selected in applications where good corrosion properties and aesthetic considerations are important. However, in cases where an austenitic SS sheet needs to be subjected to cold forming, such as deep-drawing or stamping, it exhibits a large variation in yield strength (above 50 pct) and undesirable yield strength in regions absent of deformation. The main cause of this variation is related to the cold-working process, during which the deformed regions transform from the soft parent face-centered-cubic (fcc) austenite (γ) phase into the hard martensite (α′) phase. In low-carbon austenitic SS, the tetragonality of the martensite is basically absent, and thus the martensite phase can be considered as exhibiting a body-centered-cubic (bcc) structure.

A feasible solution to resolve the variation in yield strength associated with the cold-working process has not been achieved, mainly because in the forming processes, deformation and hard martensite formation occur in areas where high strength is not required, while regions where high strength is necessary are not subjected to deformation, leaving soft austenitic regions. In these soft areas the material can be easily scratched, dented, and deformed in service, and thus its corrosion properties and appearance strongly deteriorate.

A tempting solution to this problem is to use a fully martensitic SS for the forming operation instead of an austenitic SS. In this fashion, the undeformed regions maintain the high strength and hardness of the martensite phase, whereas the deformed regions acquire the high strength and hardness associated with the martensite phase. However, due to the poor formability of the martensitic structure, products manufactured in this fashion may fracture.

To overcome the aforementioned limitations, a research plan has been designed to develop, in cold-rolled SS sheets, regions (10 to 20 mm) with nano/submicron austenite grain size, exhibiting high strength and high formability properties, while the sheet remains in the martensite phase, outside the narrow regions. Using this concept, a single SS sheet can be produced such that undeformed regions maintain the strength and hardness of the martensite, while deformed regions have enhanced strength and hardness due to the refined austenite grain structure produced within the narrow regions (Figure 1). In particular, the idea consists of heavily cold-rolling an AISI 301 austenitic SS sheet (~90 pct reduction in thickness) to induce the formation of martensite (Figure 1, step 1), followed by a local annealing treatment within the 600 °C to 900 °C temperature range to nucleate and reform the austenite phase in narrow specific regions (Figure 1, step 2) before subjecting the samples to cold forming (Figure 1, step 3). Grain sizes achieved by this method are below 1 μm.1-3 In this manner, it is possible to locally control the nano/submicron grain size and consequently the strength of the material before forming.

Within the wider scope of the aforementioned idea, the initial research objective, described in this paper, is to identify, during step 2, the influence of annealing temperature on the microstructure of a metastable austenitic AISI 301 SS. Typical factors affecting the reversion from martensite to austenite are the chemical composition, amount of cold working, annealing temperature, and dwelling time. Although previous experiments on Fe-Cr-Ni steels and plain carbon

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The goal of this paper is thus to correlate the annealing temperature with the reverted austenite grain size, phase fraction, and microstructure achieved in a commercial AISI 301 SS via magnetic measurements, X-ray diffractometry, and transmission electron microscopy (TEM) experiments. A commercial AISI 301 metastable SS grade was selected because its metastable austenitic structure facilitates the formation of stress-induced martensite and subsequently promotes an efficient austenite reversion. In addition, AISI 301 SS grades are commercially available, thereby widening the potential impact of this research work.

II. EXPERIMENTAL PROCEDURE

A. Materials

The AISI 301 SS used in this work was provided by Outokumpu Stainless Oy, Finland, with an alloy composition (wt pct) given in Table I. This SS has an Ms temperature of 18 °C\[^{[6,9]}\] and a calculated Ms temperature of −118 °C\[^{[10,11]}\]. The material was produced by continuous casting and subjected to hot rolling, heat treatment, and a final 90 pct cold-rolling reduction at Outokumpu Stainless Oy. The amount of retained δ-ferrite was found to be approximately 0.2 vol pct. As-received 90 pct cold-rolled sheets were cut into rectangular specimens, subsequently subjected to a heating rate of 100 °C/min, and annealed isothermally for 30 minutes at the temperatures 600 °C, 650 °C, 700 °C, 750 °C, 800 °C, 850 °C, and 900 °C, followed by forced air-cooling. For comparison, one specimen was left in the as-cold-rolled (CR) state.

B. Methods

1. X-ray diffraction

Rectangular sections approximately 5 × 7 × 0.8 mm\(^3\) were cut with the rolling direction (RD) parallel to the long dimension. These samples were subsequently measured in a Phillips PW1720 X-Ray Diffractometer (XRD) operating at a voltage of 40 keV and a current of 40 mA. The tests were performed at ambient temperature using CuK\(_\alpha\) radiation. Peak measurements were taken from 30 to 90 deg (2\(\theta\)) in steps of 0.1 deg with a dwell time of 4 seconds. From the resulting intensity vs 2\(\theta\) plots, MDI Jade 6.5 software was used to index the peaks. The results were compared to published XRD data for an austenitic SS\[^{[12]}\].

In addition, the X-ray spectrum of the CR specimen was used to calculate the volume fraction of austenite and martensite in this sample, based on the method published by Dickson\[^{[13]}\]. According to this method, the volume fractions of austenite C\(_\gamma\) and martensite C\(_\alpha\) were evaluated on the basis of the first three reflexions for the austenite ((111), (200), (220)) and the martensite phase ((110), (200), (211)), in the form:\[^{[13]}\]

\[
\frac{C_\gamma}{C_\alpha} = \frac{1}{n_\gamma} \sum_{i=0}^{n_\gamma} \left( \frac{I_{\gamma_i}}{R_{\gamma_i}} \right)
\]

Table I. Chemical Composition of AISI 301 Stainless Steel

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>N</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.096</td>
<td>16.7</td>
<td>6.6</td>
<td>0.0635</td>
<td>1.23</td>
<td>1.18</td>
<td>0.72</td>
</tr>
<tr>
<td>P</td>
<td>Cu</td>
<td>Co</td>
<td>S</td>
<td>D</td>
<td>Y</td>
<td>Fe</td>
</tr>
<tr>
<td>0.26</td>
<td>0.17</td>
<td>0.11</td>
<td>0.001</td>
<td>0.0031</td>
<td>0.005</td>
<td>Balance</td>
</tr>
</tbody>
</table>

where C\(_\alpha\) = I\(_\alpha\)/[1 + (C\(_\gamma\)/C\(_\alpha\))], C\(_\gamma\) = (C\(_\gamma\)/C\(_\alpha\))/[1 + (C\(_\gamma\)/C\(_\alpha\))], n\(_\gamma\) and n\(_\alpha\) are the number of martensite and austenite reflexions considered, I\(_\alpha\) and I\(_\gamma\) are the integrated intensities of martensite and austenite for the reflexions considered, and R\(_\gamma\) and R\(_\alpha\) are the relative intensities of martensite and austenite for the reflexions considered. On the basis of Eq. [1], the volume fraction of martensite present in the CR (90 pct reduction in thickness) sample can be calculated as 97.4 pct. This sample was selected as the reference value for the determination of phase fraction in all the samples by