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Materials Science and Engineering A

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# Fatigue behavior of ultrafine-grained and coarse-grained Cr–Ni austenitic stainless steels

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#### ARTICLE INFO

Article history: Received 19 September 2010 Received in revised form 25 January 2011 Accepted 26 January 2011 Available online 2 February 2011

Keywords: Austenitic stainless steel Fatigue strength Ultrafine-grained microstructure Grain boundary cracking Dislocation structure

#### ABSTRACT

High-cycle bending fatigue behavior of an ultrafine-grained Cr–Ni Type 301LN austenitic stainless steel, obtained by reversion annealing, was investigated and compared to that of the conventional coarse-grained counterpart. The fatigue limit was significantly increased from 350 MPa to 630 MPa, reaching 59% of the tensile strength, a behavior attributed to grain refinement. Fatigue cycling resulted in hardness increments that were very different between these structures; in the coarse-grained steel 47%, but only 6–10% in the ultrafine-grained steel. The fatigue damage was observed to occur by grain boundary cracking in the ultrafine-grained steel, while mostly by slip band formation and crack propagation along these slip bands and grain boundaries in the coarse-grained counterpart. Dislocation structures observed reflected these pronounced differences in the cyclic behavior.

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#### 1. Introduction

The reversion annealing of strain-induced martensite has been shown to be an efficient method for refining the grain size in metastable austenitic Cr-Ni alloys. Already two decades ago, Tomimura et al. [1] and Takaki et al. [2] used this technique for special alloys, and later it was applied to commercial austenitic stainless steel grades such as Types 304L, 316L, 301 and 301LN [3-9]. This simple route involves severe cold deformation of austenite to produce and deform strain-induced martensite, followed by short annealing to revert the deformed martensite into ultrafine/nanograined austenite. Excellent combinations of static mechanical strength and ductility have been obtained at the laboratory scale [5–9]. Furthermore, the corrosion resistance is superior than that of the cold-rolled steel [10]. More recently, favorable cellular pre-osteoblasts response to nano-grained 301LN has been observed [11,12], suggesting applications as an advanced biomaterial for medical applications.

In addition to static strength, fatigue resistance is also one of the key properties concerning the practical engineering utilization

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of ultrafine-grained (UFG) materials, in biomedical applications in surgery, among others. Fatigue behavior of ultrafine-grained materials has been extensively studied intensively in recent years. An overview on mechanical properties under monotonic and cyclic loading was presented by Höppel et al. [13] pointing out the importance of fatigue resistance for ultrafine-grained materials for potential engineering applications. A recent review concerning ultrafine-grained light alloys was presented by Estrin and Vinogradov [14]. It has been observed that the fatigue limit is significantly enhanced on grain refinement to sub-micron scale (e.g. [15]). Mabuchi et al. [16] demonstrated significantly (~30%) improved fatigue resistance of the ultrafine grain surface layer of a C–Mn steel (s.c. SUF steel) compared to that of the conventional coarse-grained structure in the mid-thickness region.

The fatigue behavior of UFG austenitic stainless steels has been investigated quite scarcely. Di Schino et al. [17,18] studied the influence of grain refinement between 1 and 50  $\mu$ m on the fatigue strength of Type 304 and high-N Cr–Mn steels. The grain refining had a strong effect on the fatigue strength of Type 304 steel, but only a small effect in the Cr–Mn–N steel due to the formation of slip bands promoted by nitrogen alloying.

There are studies showing severe deformation techniques improving fatigue strength of materials, especially in the high-cycle regime (e.g. [19,20]). In connection with the ultrasonic surface attrition technique Roland et al. [21] investigated Type 316L steel and observed  $\sim$ 21% improvement in the fatigue limit. Similarly, Uusi-

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talo et al. [22] reported very significant improvement ( $\sim$ 43–80%) in the bending fatigue resistances of Type 316L and 301LN steels after the attrition treatment. However, severe plastic deformation generates residual stresses in the material and therefore corrosion-performance of stainless steels will be impaired [10]. On the contrary, the reversion annealing can produce bulk material of homogeneous UFG structure without internal stresses.

Preliminary studies on the effect of grain size on the fatigue behavior of Type 301LN steel indicated that the fatigue mechanism is sensitive to grain size [23]. In the present paper, an extended study is presented to better understand the fatigue behavior of a phase reversion annealed UFG austenitic stainless steel, with excellent static mechanical properties with the hope to expand its potential application fields.

#### 2. Experimental procedure

The experimental material was commercial, annealed Type 301LN Cr–Ni austenitic stainless steel of 3 mm and 5 mm thick sheets. The chemical composition was (in wt.%): Fe–0.017C–1.29Mn–17.3Cr–6.5Ni–0.15Mo–0.15N. The coarsegrained (CG) 301LN specimens were taken from the 3 mm sheet. The 5 mm sheet was cold rolled to 50% total thickness reduction in a laboratory rolling mill. The strain-induced  $\alpha'$ -martensite fraction was measured by a Ferritescope instrument (Fischer Model FMP30) and was ~87% in the cold-rolled steel.

Strips of dimensions 120 mm  $\times$  30 mm  $\times$  2.5 mm were cut from the cold-rolled sheet in the rolling direction for annealing experiments on a Gleeble 1500 simulator. The heating rate was 200 °C/s to the holding temperature of 800 °C and the soaking time 10 s, based on previous results to obtain a highly refined structure [6–8]. The uniform temperature zone in the middle of the samples was estimated to be about 25 mm in length.

A field emission gun scanning electron microscope FEG-SEM (Carl Zeiss Ultra plus) applying electron backscatter diffraction (SEM-EBSD) was applied to determine the average grain size. In EBSD scans, the acceleration voltage of 20 kV and a step size of 0.03  $\mu$ m were used. For examinations, the specimen surfaces were mechanically polished down to 1  $\mu$ m by using a diamond suspension, and then chemically polished by a 0.05  $\mu$ m colloidal suspension of silica for about 20 min. Overall, about 2000 individual grains were measured, excluding low angle boundaries (the misorientation below 15°) for the grain size. The grain-EBSD maps were processed by applying the grain reconstruction analysis in HKL-Channel 5 software.

The tensile tests were carried out at room temperature at the constant strain rate of  $10^{-3}$  s<sup>-1</sup>. Test specimens machined from the annealed strips were with the gage section of 2 mm × 6.25 mm and the gage length of 20 mm. The fatigue tests were carried out in air at room temperature using a bending fatigue machine driven at a frequency of 23 Hz with the zero mean stress. The standard hourglass-shape of flat fatigue specimens with the thickness 2 mm were used in all tests, similarly as in previous studies [23–25]. The specimens were ground to obtain smooth surfaces and polished mechanically using the diamond suspension of 3  $\mu$ m size to obtain mirror finish.

Hardness of some specimens was measured after cycling to failure employing either the conventional Vickers diamond pyramid method (macrohardness using 5 kg load, HV5) or by microhardness method using a Berkovich tip with a load of 100 mN.

The cyclic damage and crack formation were examined using an optical microscope (OM), a FEG-SEM applying either the electron channeling contrast (SEM-ECC) imaging or SEM-EBSD, and transmission electron microscopy (Hitachi 7600) operated at 120 kV. Thin foils were prepared using a twin-jet electropolishing at a volt-



**Fig. 1.** Grain structure of (a) as-received coarse-grained (CG) sheet (OM) and (b) 50% cold-rolled steel after reversion annealing at 800 °C for 10 s (SEM-EBSD).

age of 25 V at a temperature of  $10 \,^{\circ}$ C. The electrolyte contained  $10 \,\text{vol.}\%$  of perchloric acid and 90% of acetic acid.

#### 3. Results

#### 3.1. Microstructure and grain size

The grain structure of the as-received annealed CG 301LN steel with fully austenitic structure and the grain size of  $\sim 20 \,\mu$ m is presented in Fig. 1a. Upon annealing at 800 °C for 10 s, the strain-induced martensite (the fraction about 87%) in the 50% cold-rolled sheet reverted to UFG austenite via the diffusional process, as confirmed in our earlier studies [6–8]. Fig. 1b shows the typical microstructure of the reversion-annealed sheet. The microstructure consists of a mixture of ultrafine reverted austenite grains; the average size 0.75  $\mu$ m; along with a few larger retained austenite grains, which did not transform to martensite during the cold rolling. This presence of bimodal structure is in agreement with observations reported earlier [9]. In good consistence, Rajasekhara et al. [26] obtained fine grains of 0.8  $\mu$ m in size in the same steel, as cold rolled 63% and annealed at 800 °C for 10 s.

#### 3.2. Static properties

Static mechanical properties of the UFG and CG steels are listed in Table 1. As reported earlier in several papers, grain refinement effectively improves the static strength and excellent strength–ductility combinations can be obtained [6–8]. In our case, the yield strength (YS) was increased by almost 100%. The Hall–Petch relationship has been found to be obeyed between the UFG size and YS of 301LN [26]. Download English Version:

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