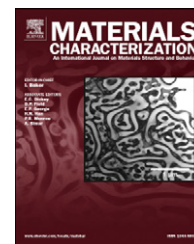


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Grain refinement of a nickel and manganese free austenitic stainless steel produced by pressurized solution nitriding



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ABSTRACT

Prolonged exposure at high temperatures during solution nitriding induces grain coarsening which deteriorates the mechanical properties of high nitrogen austenitic stainless steels. In this study, grain refinement of nickel and manganese free Fe–22.75Cr–2.42Mo–1.17N high nitrogen austenitic stainless steel plates was investigated via a two-stage heat treatment procedure. Initially, the coarse-grained austenitic stainless steel samples were subjected to an isothermal heating at 700 °C to be decomposed into the ferrite + Cr₂N eutectoid structure and then re-austenitized at 1200 °C followed by water quenching. Microstructure and hardness of samples were characterized using X-ray diffraction, optical and scanning electron microscopy, and micro-hardness testing. The results showed that the as-solution-nitrided steel decomposes non-uniformly to the colonies of ferrite and Cr₂N nitrides with strip like morphology after isothermal heat treatment at 700 °C. Additionally, the complete dissolution of the Cr₂N precipitates located in the sample edges during re-austenitizing requires longer times than 1 h. In order to avoid this problem an intermediate nitrogen homogenizing heat treatment cycle at 1200 °C for 10 h was applied before grain refinement process. As a result, the initial austenite was uniformly decomposed during the first stage, and a fine grained austenitic structure with average grain size of about 20 μm was successfully obtained by re-austenitizing for 10 min.

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1. Introduction

Nitrogen has been successfully used as an inexpensive alloying element to develop commercial high nitrogen austenitic stainless steels (HNASSs) with high strength and high corrosion resistance [1–4]. Nitrogen is a strong austenite stabilizer and therefore, it can be used as a substitute for the expensive nickel. Additionally, the nickel contained in the conventional medical stainless steels (for example AISI 316L grade) causes allergy problems in the human body. Therefore, production of nickel free HNASSs offers a new perspective on

the development of more efficient metallic biomaterials [5–8]. During the last decades, different grades of Fe–Cr–Mn–Mo–N [9–12] and Fe–Cr–Mn–N [13,14] Ni-free HNASSs have been developed as new biomedical steels. These high nitrogen stainless steels are produced by high pressure melting under nitrogen atmosphere and contain large amount of chromium (15–25 wt.%), manganese (10–24 wt.%) and nitrogen (0.45–1.1 wt.%). However, recent studies in cytotoxicity evaluation of metal salts using murine fibroblasts and osteoblastic cells have indicated rather high toxicity of manganese and their salts [15,16]. Thus, more recently nickel and manganese free

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(Ni- and Mn-free) austenitic stainless steels have drawn attention as harmless biomaterials [17].

Recently, solution nitriding (also called high temperature gas nitriding) was recognized as a versatile, simple and promising solid state manufacturing process for fabrication of the high nitrogen stainless steels. The process, which was initially introduced by Berns et al. [18], runs out at high temperatures above 1050 °C under nitrogen gas atmosphere. Using this process, it is possible to achieve up to 2.5 mm nitrided depths on stainless steels [19–22]. This enables the process to be used for nitriding the whole thin sections. Thin sectioned components could be fabricated into their final shape in the ferritic state and then austenitized using solution nitriding process. Therefore, nickel and manganese free austenitic stainless steel plates, small devices and foils with high strength and corrosion resistance can be fabricated by nitriding the Ni- and Mn-free ferritic stainless steel under N₂ gas at 1000–1200 °C without requiring any special equipment.

However, prolonged exposure at high temperatures during solution nitriding process results in the formation of coarse-grained austenite with inferior mechanical properties. This unfavorable effect becomes more important with increasing the sample thickness. On the other hand, most of the Ni- and Mn-free HNASSs (including Fe–25Cr–1.1N [23], Fe–24Cr–N and Fe–24Cr–2Mo–N [24] and Fe–22.75Cr–2.42Mo–N [25] steels) are inherently susceptible to inter-granular brittle fracture in tensile testing at ambient temperature. The research studies performed using Vickers indentation tests [25] and Transmission Electron Microscopy (TEM) [23] imply that the inter-granular brittle fracture is closely related to activation of straight slip bands and formation of pile ups with planar array of dislocations at the grain boundaries. To suppress inter-granular fracture, the stress concentration exerted by planar array of dislocations at the grain boundaries should be decreased. Therefore, application of a subsequent grain refinement process was proposed as a route to achieve this purpose.

The common process of grain refinement via plastic deformation cannot be applied on Ni- and Mn-free HNASSs due to low ductility despite of having Face Centered Cubic (FCC) structure [25,26]. In contrast, the phase diagram of the Ni- and Mn-free HNASSs predicts a heat treatment route for austenite grain refinement. When Ni- and Mn-free high nitrogen austenite is isothermally heated below the austenitizing temperature, it becomes unstable and decomposes to ferrite and chromium nitride (i. e. $\gamma \rightarrow \alpha + \text{Cr}_2\text{N}$). If fully decomposed high nitrogen austenitic steel is again reheated to the austenitizing temperature and held for a sufficient time, the structure will transform to austenite. As many nucleation sites are available, the size of the new austenite grains will be small.

The production of the nickel and manganese free austenitic stainless steels by solution nitriding has many advantages and is regarded as promising route. However, more investigations are needed to be done in order to overcome the grain coarsening and brittle fracture problems and also to deeply understand the austenite grain refinement process. It seems that post-grain refinement treatment is a useful way to improve the tensile properties and fracture toughness. The present study is aimed at investigating the grain refinement of a Ni- and Mn-free Fe–22.75Cr–2.42Mo–1.17N austenitic

stainless steel plates, produced by solution nitriding at 1200 °C in a nitrogen gas atmosphere under pressure of 0.25 MPa. The austenite grains were refined by a two-stage isothermal (decomposition–re-austenitizing) heat treatment procedure. To achieve successful grain refinement, a homogenization pre-heat-treatment was applied and its effects on grain refinement process of the steel were investigated and discussed.

2. Experimental Procedure

Nickel and manganese free Fe–22.75Cr–2.42Mo–1.17N austenitic stainless steel plates with 2 mm thickness were produced by solution nitriding at 1200 °C under nitrogen gas pressure of 0.25 MPa. The Fe–Cr–Mo alloy was prepared by induction melting in an argon atmosphere followed by electroslag remelting and homogenizing at 1100 °C for 48 h. Table 1 indicates the chemical composition of the investigated Fe–Cr–Mo steel in this research. The initial ferritic plates were prepared by hot rolling of a 10 mm thick slab at 1000 °C. The plates were annealed at 900 °C for an hour before solution nitriding.

Nitrogen was added to ferritic stainless steel plates by solution nitriding in horizontal tube furnace. Samples were ultrasonically cleaned successively in acetone and ethanol and were located in the AISI 304 steel stages before high temperature gas nitriding process. The air in the furnace was purged with nitrogen for 15 min to prevent oxidation of the samples. After the purging was completed, the heating procedure at 10 °C/min was started and nitrogen gas was introduced into the furnace continuously. When the temperature reached 1200 °C, the pressure of nitrogen gas raised to maintain a pressure of 0.25 MPa (2.5 bar) in the furnace. Finally, the nitrided specimens were water quenched. The optimum full nitriding time was obtained based on the solution nitriding at different times of 1, 3, 6, 9, 12, 13, and 18 h. At prolonged nitriding times, chromium nitride precipitates are formed via a discontinuous precipitation mechanism [25].

Coarse grains of the as-solution-nitrided steel were refined by a two-stage isothermal heat treatment procedure. Microstructural investigations were performed using optical microscopy (OM, Olympus-PMG3) and scanning electron microscopy (SEM, JSM-5900 operated at 20 kV). The polished surface was chemically etched in modified aqua regia (50% HCl–25% HNO₃–25% H₂O (vol.%)). Phase composition of the samples was studied by X-ray diffraction (XRD) using a Bruker D8 advanced diffractometer operated at 40 kV and 40 mA with the Cu-K_α radiation ($\lambda = 1.5406 \text{ \AA}$) in the Bragg–Brentano configuration. XRD patterns were recorded with step size of 0.05° and step durations of 2 s at each step in the angular range of 40°–100°.

The average nitrogen content in the ferritic and austenitic samples was measured using a LECO TN500 nitrogen determinator in a self-contained electrode furnace for fusion. In this method the nitrogen content is determined by measuring the thermal conductivity of the nitrogen containing gas in a Thermal Conductivity (TC) cell. The cross-sectional hardness was investigated using an MDPEL-M400 GL microhardness tester equipped with a Vickers indenter. Micro-hardness measurements were performed under the loads of 100 and

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