SUBMICROCRYSTALLINE STRUCTURE AND INTER-GRANULAR CORROSION OF AN AUSTENITIC STAINLESS STEEL SUBJECTED TO MULTIDIRECTIONAL FORGING AND ANNEALING

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Abstract

The corrosion properties of the ultrafine grained S304H austenitic stainless steel samples subjected to severe plastic deformation and annealing are investigated. The severe plastic deformation is carried out by means of multidirectional forging at different temperatures ranging from 500 to 800°C. The influences of processing conditions on the developed microstructure and the resistance to intergranular corrosion are evaluated.

Keywords:

austenitic stainless steel, severe plastic deformation, ultrafine grained structures, intergranular corrosion

Introduction

Austenitic stainless steel is one of the most frequently used types of structural metallic materials. However, its applications as a construction material are highly restricted by low strength. The problem of increasing the strength of austenitic steels can be solved by reducing the size of structural elements, grains and subgrains (Valiev, 2010). Severe plastic deformation (SPD) is one of the most effective ways to get a submicrocrystalline structure in steels and alloys. When selecting a mode of SPD for increasing strength properties, you must consider two parameters which can affect ~~on~~ corrosion properties of steel. First, the higher the density of the boundaries and dislocations, the lower the resistance to intergranular corrosion (IGC). Secondly, allocation of particles, enriched with chromium at the SPD temperature may have an adverse affect on corrosion resistance. The aim of this work is to study the effects of temperatures of SPD and annealing on the structural parameters and resistance to IGC of an S304H austenitic stainless steel.

Experimental Procedure

An S304H austenitic stainless steel, 0.10%C–18.2%Cr–7.85%Ni–2.24%Cu–0.50%Nb–0.008%B–0.12%N–0.95%Mn–0.10%Si and the balance Fe (all in weight%), with an average grain size of about 7 μm is used as the starting material. Rectangular samples are subjected to multidirectional forging (MDF), which is carried out using isothermal multi-pass compression tests with a change in the compression direction in 90° in order of three orthogonal axes from pass to pass at temperatures of 500, 600, 700, 800°C. A total strain of 4 was applied for each sample. Then, the MDF samples were annealed at temperature of 800°C for 30 minutes followed by water quenching.

The structural investigations are carried out on the sample sections parallel to the forging direction in the last pass by using an optical microscope, a Jeol JEM-2100 transmission electron microscope (TEM) and a Quanta 250 Nowa scanning electron microscope (SEM) equipped with an electron back scattering diffraction (EBSD) analyser incorporating an orientation imaging microscopy (OIM) system. The intergranular corrosion resistance is evaluated by a double loop electrochemical potentiokinetic reactivation (EPR) tests (Majidi and Streicher, 1984) using a 0.5 M H2SO4 + 0.01 M KSCN electrolyte at 30±1°C and a scan rate of 3 mV/s. The calculation of the current density is made in the program IPC-Compact-M.

1. Deformation and annealed microstructures

1.1 Deformation microstructures

Typical deformation microstructures developed after ten forging passes at temperatures of 500, 600, 700, and 800°C are shown in Figure 1.

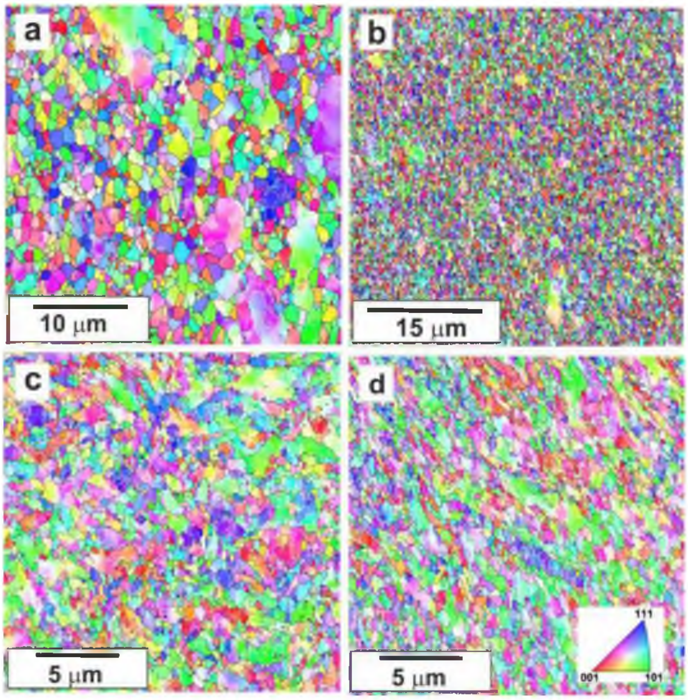

Fig. 1: Microstructure of S304H steel after MDF to $\varepsilon = 4$ at: a) 800°C, b) 700°C, c) 600°C, d) 500°C

The uniform ultrafme grained structures are evolved in the samples processed at 800 and 700°C. The deformation microstructures are almost fully composed of nearly equiaxed ultrafine gains, which are entirely delimited by high angle boundaries and are considered to be dynamically recrystallized grains. The average size of these grains in the sample processed at 800°C is 0.69 μm (Tikhonova, 2012). In the sample processed at 700°C, the average size of dynamically recrystallized grains is 0.3 μm, and their volume fraction comprises 0.87.

At temperatures of 500°C and 600°C, the deformation microstructures are characterized by vague heterogeneities. These microstructures contain relatively large grains with irregular boundaries in addition to the equiaxed ultrafine dynamically recrystallized grains. Evidently, grains with such rough appearances represent the remainders of original grains. Commonly, the size of ultrafine dynamically recrystallized grains and their volume fraction decrease with a decrease in the deformation temperature. The size of ultrafine grains and their volume fraction comprise 0.2 μm and 0.67 at 600°C, respectively, and 0.1 μm and 0.51 at 500°C.

1.2 Annealed microstructures

Typical microstructures evolved in the S304H austenitic stainless steel subjected to MDF at 500 and 800°C and then annealed at 800°C during 30 min are shown in Figure 2.

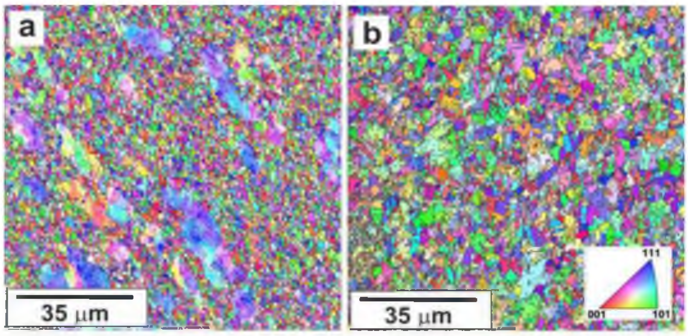

Fig. 2: Microstructures of S304H steel after MDF to ε = 4 at: a) 800°C, b) 500°C- and annealed at 800°C

The samples processed by MDF at relatively high temperature of 800°C are quite stable against grain coarsening during the subsequent annealing at 800°C. The grain size of 0.69 μm that evolved by MDF slightly increases to 0.78 μm after 30 min annealing. On the other hand, the 30 min annealing results in twofold increase in the mean grain size from 0.3 to 0.68 μm in the sample subjected to MDF at 600°C. Some microstructural inhomogeneity should also is noted for this sample. Namely, rather large annealed grains appear as islands surrounding by much fine grains.

2. Intergranular corrosion

2.1 Effect of MDF temperatures

The MDF at different temperatures leads to the formation of ultrafine grained structure in steel S304H (Tikhonova, 2013). Moreover, the average grain size decreases with decreasing deformation temperature. The IGC resistance can be evaluated by a ratio of Ir/Ia, where the Ia is the activation peak current of the anodic scan and the Ir is the reactivation peak current in the reversed scan (Kina, 2008). The values of Ir/Ia ratio for the S304H samples subjected to MDF at different temperatures are shown in Figure 3.

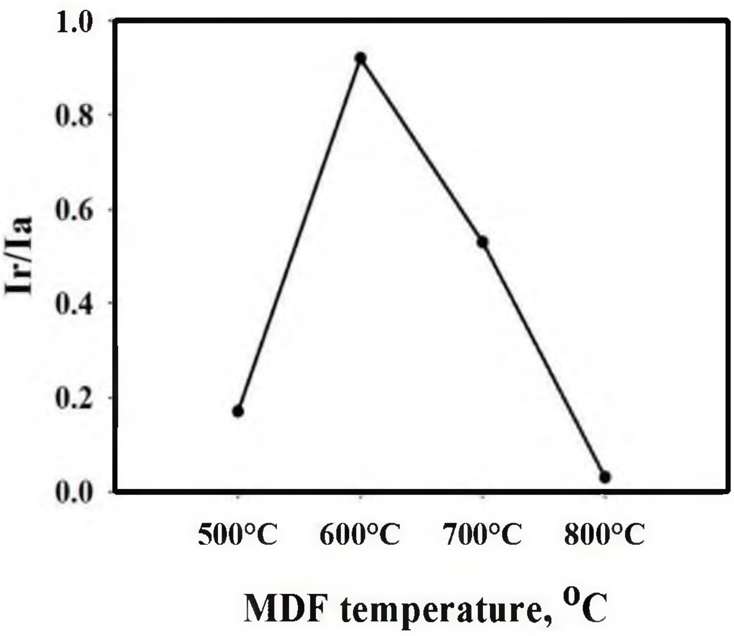

Fig. 3: The ratios of Ir/Ia for the S304H steel samples subjected to MDF at different temperatures

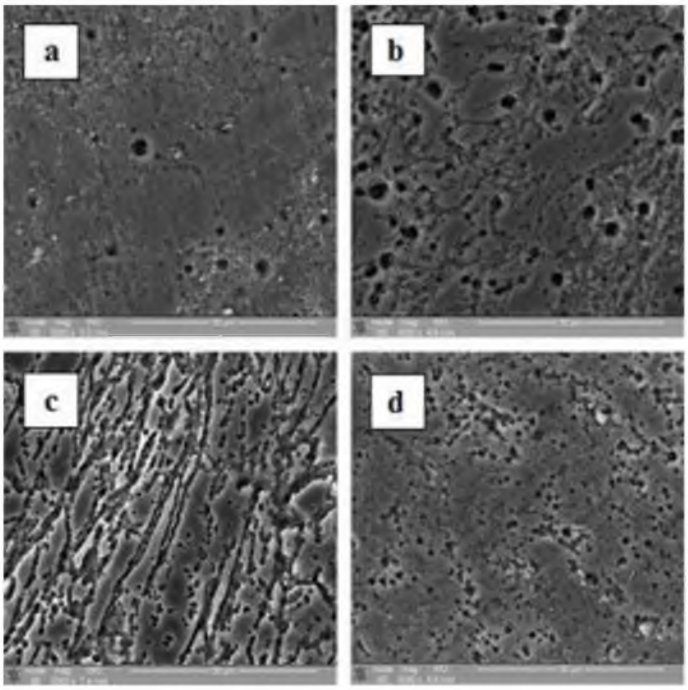

Fig. 4: The surface of S304H steel samples after IGC testing: a) MDF at 800°C, b) MDF at 700°C, c) MDF at 600°C, d) MDF at 500°C

In the sample subjected to MDF at temperature of 800°C, the ratio of Ir/Ia does not exceed the value of 0.11. This indicates the resistance of steel against the IGC. The smooth surface of this sample after IGC testing (Fig. 4a) also suggests the good corrosion resistance. The ratios of Ir/Ia in the samples subjected to MDF at temperatures of 500-700°C remarkably exceed a critical value of 0.11, which is considered as the required limit for corrosion resistant steels (0.11). It should be noted that the sample subjected to MDF at temperature of 600°C is the most succeptible to IGC; the Ir/Ia is nearly 9 times higher than the required value. The surface of this sample after IGC testing is highly damaged by corrosion (Fig. 4c).

It is known that the formation of $Cr_{23}C_6$ particles takes place in 304itype steels during annealing at temperatures of 450-700°C. The calculation results of the volume fraction of chromium carbide for the steel S304H by the Thermo-Calc software is presented in Table 1. The highest volume fraction of carbides is obtained at temperature of 600°C. Therefore, the depletion of grain boundaries with chromium may lead to a low IGC resistance in the samples processed at this temperature (Roncerya, 2011).

Table 1: Calculation of volume fraction of $Cr_{23}C_6$ at different temperatures in S304H steel by Thermo-Calc

Temperature,°C	500	600	700	800
Volume fraction $Cr_{23}C_6$	0,0198	0,02	0,0198	0,018

2.2 Effect of Annealing

The steel samples subjected to MDF and then annealed at 800°C exhibit quite low susceptibility to IGC. The ratio of Ir/Ia in these annealed samples is almost zero. The excellent resistance of these samples to IGC is also suggested by smooth sample surfaces after corrosion tests as revealed by SEM observation (Fig. 5). Thus, the IGC isn't observed in the annealed samples.

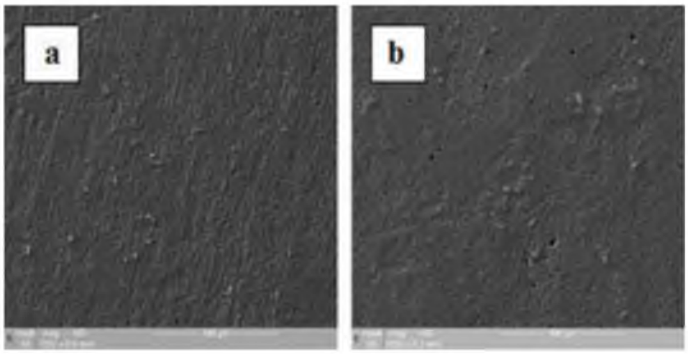

Fig. 5: The surface of S304H steel samples after IGC testing: a) MDF at 800°C followed by annealing at 800°C, b) MDF at 500°C followed by annealing at 800°C

The absence of reactivation peaks at DL-EPR curves (Fig. 6) testifies to IGC resistance.

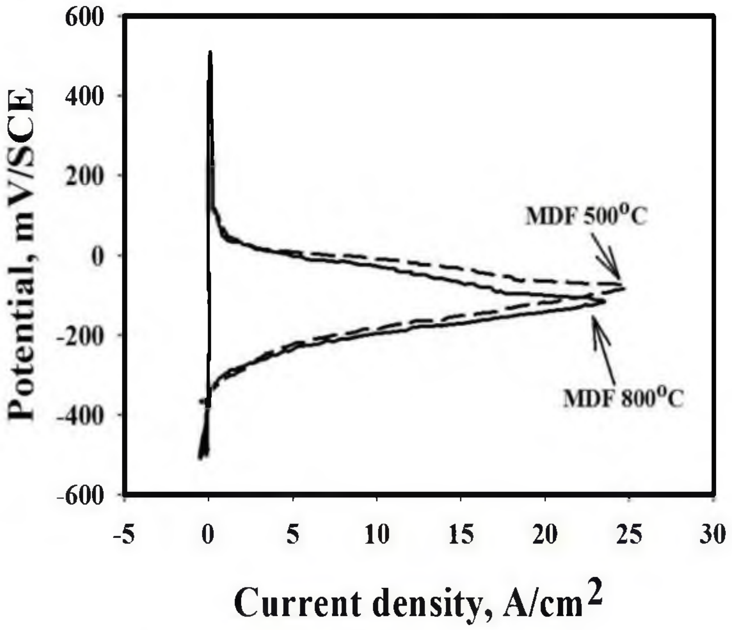

Fig. 6: Double loop electrochemical potentiokinetic reactivation curves of samples subjected to MDF at 500, 800°C and then annealed at 800°C

Annealing of the ultrafine grained steel samples processed by warm severe plastic deformation significantly increases their corrosion resistance. This annealing effect on the corrosion resistance may be caused by various reasons. Both the development of equilibrium grain boundaries instead of non-equilibrium strain-induced ones and an increase in the grain size during annealing may promote the corrosion resistance. Also, an annealing at elevated temperatures removes the Cr-depleted zones along grain boundaries and equalizes the chemical composition throughout the sample, suppressing the susceptibility to IGC.

Summary

The influence of the MDF temperature and annealing temperature on the IGC resistance of the ultrafine grained S304H stainless steel is presented. The sample processed by MDF at 800C exhibit the highest resistance to IGC. In contrast, the sample subjected to MDF at 600C is the most susceptible to IGC. Annealing of the ultrafine grained steel samples at 800C during 30 min makes the samples unsusceptible to IGC.

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