Materials Science & Engineering A 734 (2018) 85–92

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Precipitation and mechanical properties of UNS 2205 duplex steel subjected to hydrostatic extrusion after heat treatment

P. Maj[a,*], B. Adamczyk-Cieslak[a], J. Nowicki[a], J. Mizera[a], M. Kulczyk[b]

[a] Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska 141, 02-507 Warsaw, Poland
[b] High Pressure Research Center, Polish Academy of Sciences, Sokołowska 29, 01-142 Warsaw, Poland

ARTICLE INFO

Keywords:
Hydrostatic extrusion
Duplex steel
Precipitation
Mechanical properties
Heat treatment

ABSTRACT

The aim of this study was to analyze the impact of severe plastic deformation on 1.4462 duplex stainless steel (UNS 2205) subjected to hydrostatic extrusion (HE) and subsequent heat treatment at selected temperatures. The tests were conducted on a material deformed with a total strain of $\varepsilon = 3.8$ after annealing in the temperature range of 400–800 °C. The characterization was carried out using transmission electron microscopy (TEM), optical microscopy, Vickers hardness and tensile testing. The results showed that the strength of the steel was a function of strain aging, grain growth and phase transformation. The highest Ultimate Tensile Strength measured in the experiment was nearly 2.1 GPa compared with 1.7 GPa for the cold-deformed material. The changes in the austenite and ferrite phases after annealing were different, resulting in a peculiar microstructure with a mix of very small and fragmented γ and σ phases and recrystallized α grains. This resulted in an increase in its strength and a bimodal structure. Overall the research has described in detail the interactions that are present in high alloy steel during the heat treatment process.

1. Introduction

Severe Plastic Deformation (SPD) describes a large variety of metalworking techniques that can be used to obtain ultrafine grains by applying large strains. This is possible thanks to special tooling which can induce a complex stress state or high shear stress which inhibits the creation of cracks during the manufacturing process. The most common methods encountered in the literature are Equal Channel Angular Pressing (ECAP) [1,2], High Pressure Torsion [3,4], and Accumulative Roll Bonding (ARB) [5,6]. All of the techniques mentioned have their advantages and disadvantages, notably the small number of baths, the heterogeneity of deformation of the material and the low productivity. On the other hand, they all have the ability to obtain very small grain sizes that significantly change the mechanical properties and microstructure of the material [7,8]. Nevertheless, due to the numerous drawbacks, they are still not commonly used in the industry.

Hydrostatic Extrusion (HE) is a method which has been developed at the Institute of High Pressure Physics (Poland)[9,10]. The input metallic material takes the form of a rod. The whole process is carried out in a sealed cylinder that contains a hydrostatic medium that exerts pressure on the billet extruding the metal through a die. This process has several advantages, among others the frictionless movement of the material. This allows for faster speeds, higher reduction ratios and enhanced cooling which prevent grain growth [11,12].

There are numerous works concerning the HE process and its effect on the microstructure and mechanical properties of many different metals. It should be noted that relatively high strength material and difficult to deform materials can be processed in this way. Especially materials with low stacking fault energy (SFE), as they seem to be more prone to grain refinement. On the one hand after achieving a certain relatively low degree of total strain the deformation mechanism tends to change, hampering further refinement. On the other hand, high SFE materials will undergo constant changes that will accumulate with successive stages of deformation. From a scientific viewpoint it is interesting how both mechanisms interact with each other, creating complex microstructures. An example of such interactions can be observed in duplex steels where the austenite phase has an energy of 11 mJm^{-2} [13] and the ferrite phase 380 mJm^{-2} [14]. The authors previously studied UNS 2205 (1.4462) stainless steel after HE with a total strain of $\varepsilon = 3.8$. The results were analyzed in a previous article [15] from the viewpoint of microstructural changes and its mechanical properties. There was a different structural response of both phases to high levels of deformation. The austenite phase reached a saturation of deformation relatively early in the process, whereas the changes in the ferrite phase were more gradual. Furthermore, at the highest degree of deformation a non-diffusive (martensitic) transformation γ→α'

* Corresponding author.
E-mail address: piotr.maj@pw.edu.pl (P. Maj).

https://doi.org/10.1016/j.msea.2018.07.020
Received 16 March 2018; Received in revised form 29 May 2018; Accepted 6 July 2018
Available online 29 July 2018
0921-5093/

Table 1
Phases occurring in high alloy stainless steels [19].

Phase	Nominal chemical formula	Temperature range [C]	lattice parameter, nm
ferrite	…	…	0.286–0.288
austenite	…	….	0.358–0.362
σ	Fe-Cr-Mo	600–1000	a = 0.879, c = 0.434
Chromium nitride	Cr2N	700–900	a = 0.480, c = 0.447
Chromium nitride	CrN	…	a = 0.413–0.447
X	Fe36Cr12Mo10	700–900	0.892
R	Fe-Cr-Mo	550–650	a = 1.090, c = 1.934
π	Fe7Mo13N4	550–600	0.647
τ	ND	550–650	a = 0.405, b = 0.484, c = 0.286
M7C3	…	950–1050	a = 0.452, b = 0.699, c = 1.211
M23C6	…	600–950	a = 1.056–1.065

occurred in the shear bands where the stress levels were the highest. The strength of the obtained material was very high, up to 1760 MPa, with a very low elongation of 2.4%. The results were very promising although there were similar research papers that had approximately the same values for the mechanical properties [16–18]. The current research has been focused on the heat treatment of stainless steel subjected to SPD. The most important achievement, to link microstructure and the mechanical changes, occurred as a result of temperature. Various factors were identified that are related to the rearrangement of the microstructure after the heat treatment and the induced strain. Additionally, new possibilities were established that can enable the homogeneous intermetallic σ precipitates to be obtained throughout the whole volume of the material.

The tested material undergoes numerous transformations during the heating process. According to the literature, there are over 13 phases that can be seen in high alloy stainless steel, α, α_{cr}, γ, σ, χ, R, π, τ, G, CrN, Cr_2N, $M_{23}C_6$ [19]. Depending on the heating temperature, time and pre-deformation, the order and kinetics of the precipitation occurrence may change. The ranges of occurrence that may be encountered in UNS 2205 stainless steel have been given in Table 1, although it should be noted that the pre-strain may decrease the formation temperature and increase the kinetics of some precipitates [3,20]. Furthermore, it should be noted that the martensitic transformation caused by severe deformation increases the ratio of the ferrite-austenite grain boundaries, which are important precipitation sites of σ and χ which will affect the overall mechanical properties of the material [21].

The overall aim of the current research was to analyze the influence of heat treatment on the mechanical properties of duplex steel after a martensitic transformation and heat treatment. The data in the literature [18,22] does not clearly predict the response of the material. Higher temperatures will lead to the reduction of the internal stresses caused by high deformation. However, according to the literature [23], strain aging is a factor which can even further increase the strength of the material. On the other hand, it is claimed that due to the σ phase the material may be susceptible to cracking [24]. However, by controlling the heat treatment parameters it is possible to enhance the mechanical properties by ensuring the even distribution of the precipitates [25]. The conducted tensile tests showed an increase of up to 2100 MPa with low elongation similar to the extruded sample. Changes in the microstructure as a function of the annealing temperature in both phases were clearly visible and had different kinetics of grain growth. What is worth noting is that equiaxed grains were seen at 800 °C but there was also a large fraction of very fine σ precipitates and high stress at the microstructural level.

Table 2
The chemical composition [wt%] of the UNS 2205 duplex steel used in the experiment, measured using the XRF method.

Cr	Ni	Mo	Mn	C	N	Fe
22.85	5.12	3.18	0.86	max 0.3	0.02 ÷ 0.08	66.83

2. Material and experimental procedure

The material used in the experiment was a commercial 1.4462 duplex stainless steel with the chemical composition as given in Table 2. The initial billet with a diameter of 20 mm was extruded at room temperature in eight consecutive steps to 3 mm diameter. The obtained material was annealed in a resistance furnace for 30 min in the range of 400–800 °C with intervals of 100 °C. The microstructure studies were done using a ZEISS Axio Scope A1 metallographic microscope.

The microscopic observations were carried out using a JEOL JEM 1200 EX II with an accelerating voltage of 120 kV. 2. The samples were cut by Wire Electrical Discharge Machining (WEDM). The 3 mm diameter discs were thinned, first by sand paper grinding and then electro polished using Struers Tenopol 5 with 10% Perchloric Acid + 90% Methanol. The equivalent grain size d_{eq} (defined as the diameter of a circle which has a surface area equal to the surface area of a given grain) was determined using a computer equipped with an image analyzer. The existing phases were identified and their volumes estimated by X-ray diffraction (XRD) analysis, performed at room temperature using a Bruker diffractometer D8 Advance, working with Co Kα radiation ($\lambda K\alpha = 0.179$ nm). The authors used the Averbach and Cohen method according to ASTM standards. The integrated intensities of the austenite phase, (200) and (220), and the ferrite phase, (200) and (211), diffraction peaks were measured producing four austenite/ferrite phase peak intensity ratios. The use of multiple diffraction peaks minimized the effects of preferred orientation providing better measurement accuracy.

3. Results

3.1. Optical microscopy

Due to the high strain, the fine microstructure in the deformed material was obtained after deformation (Fig. 1). Due to electrolytic etching in a 20% NaOH water solution the microstructure of the tested material was revealed. Ferrite regions became dark whereas the austenite regions remained mostly white. Comparing both the longitudinal and perpendicular structure to the extrusion directions it can be observed that in the longitudinal direction the grains were deformed into needle-like shapes. Whereas in the perpendicular direction the structure was very fine and uniform and did not show any special characteristics. The morphological anisotropy was very intense due to the multiple extrusions. Observations using light microscopy do not allow the phases presented in Table 1 to be distinguished due to their small size and large deformation.

4. TEM Microscopy

4.1. As-deformed state

For closer observation TEM was used for the characterization of duplex stainless steel with $\varepsilon = 3.8$. The refinement of the microstructure was confirmed, although according to the image analysis and widening of the diffraction peaks there were different responses in both the ferrite and austenite phases (Fig. 2). According to the previous investigation performed by the authors [15] the first phase tends to deform mainly by twinning, where in the second phase slip deformation is dominant. However, it should be noted that due to the high cumulative