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Microstructure and mechanical properties of duplex stainless steel subjected to hydrostatic extrusion

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ABSTRACT

The nanostructure and mechanical properties of ferritic-austenitic duplex stainless steel subjected to hydrostatic extrusion were examined. The refinement of the structure in the initial state and in the two deformation states ($\varepsilon = 1.4$ and $\varepsilon = 3.8$) was observed in an optical microscope (OM) and a transmission electron microscope (TEM). The results indicate that the structure evolved from microcrystalline with a grain size of about 4 μm to nanocrystalline with a grain size of about 150 nm in ferrite and 70 nm in austenite. The material was characterized mechanically by tensile tests performed in the two deformation states. The ultimate strength appeared to increase significantly compared to that in the initial deformation stages, which can be attributed to the grain refinement and plastic deformation. The heterogeneity observed in microregions results from the dual-phase structure of the steel. The results indicate that hydrostatic extrusion is a highly potential technology suitable for improving the properties of duplex steels.

1. Introduction

Nanomaterials are very interesting for scientists and the industry. They have many unique properties distinguishing them from conventional bulk materials. Their yield strength and ultimate strength are increased due to the large fraction of grain boundaries [1]. Their catalytic properties are also better which results from the enlarged specific surface area [2]. One of the interesting applications of nanomaterials is the manufacture of new magnetic materials [3].

Nanomaterials may be synthesized by consolidating small clusters, atom by atom or layer by layer, in a configuration known as bottom-up. The most common methods in this group are: inert gas condensation [4], electro-deposition [5], chemical and physical vapor deposition [6,7], and crystallization from amorphous states [8]. Another way to obtain nanomaterials is the top-down approach. This group of methods includes severe plastic deformation (SPD) [9,10], cryomilling [11], and nanolithography [12]. Thus far, the most efficient way to achieve large volumes of a nanomaterial has been the SPD process. The most common methods in this group are equal channel angular pressing (ECAP) [13,14], high-pressure torsion (HPT) [15,16], and hydrostatic extrusion (HE) [17]. The desired shape of the material is obtained by using tools of special geometries which prevent the free flow of the material and thereby generate a significant hydrostatic pressure. The large shear strains generate crystal lattice defects of high density, especially dislocations, which can lead to a significant refinement of the grains.

HE enables us to achieve a nanostructure in a large group of materials [18–21]. The process is carried out in a sealed cylinder filled with a hydrostatic medium. The piston movement ensures the appropriate pressure needed to extrude the material through the die, but there is no direct contact

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between it and the batch. Moreover, the pressure inside the cylinder acts uniformly on the whole workpiece inducing compressive stress and creating favorable forming conditions. As a result large volumes of bulk nanocrystaline material can be achieved that exceed in quanity other SPD methods. The whole process is carried out at very high deformation rates which can exceed 10^3 s^{-1}. According to the available literature, high strain rates can enhance grain refinement [1]. It is worth noting that, during extrusion, the friction between the die and the material is significantly reduced due to the hydrostatic medium and the lubricant additionally applied at the outlet of the die. This contributes to a high surface quality of the product and reduces heat generation. The additional cooling of the batch material at the outlet of the cylinder hinders grain growth after the process.

When predicting the mechanical properties of the alloys composed of two ductile phases, many factors must be taken into account. This is so because of the differing responses of these two phases to stress. The factors such as the morphology, grain boundary interactions and volume fractions play a significant role when predicting the mechanical response of the entire material. This is especially important when subjecting the material to severe deformations. Various researchers were concerned with this subject [22–24]. Promising results were obtained for ferritic-austenitic duplex steel subjected to accumulative roll bending [25] and equal channel angular pressing [26]. It is known from these studies that the deformation in the material is heterogeneous [27,28]. Stronger strain hardening occurs in austenite and this enforces more strain accommodation in ferrite [29]. In the early stages of deformation, austenite undergoes rapid grain subdivision caused by twinning and the generation of dislocations [30]. All these factors play a significant role in the HE process.

The aim of the present study was to investigate the effect of HE on the microstructure and mechanical properties of duplex stainless steel.

2. Material and Experimental Procedure

The material used in the investigations was a commercial 1.4462 duplex stainless steel with the chemical composition (wt.%) as given in Table 1.

The initial billet with a diameter of 20 mm was extruded at room temperature in eight consecutive steps. The process parameters are given in Table 2. The process was performed at the Institute of High Pressure Physics "UNIPRESS" (Poland).

The microstructural evolution during HE process was investigated using a NIKON EPIPHOT 200 optical microscope.

Table 1 – The chemical composition of 1.4462 duplex steel [wt.%].

	C	Mn	Cr	Ni	Mo	N	Fe
				[wt.%]			
Max	0.3	2.0	23	6.5	3.5	0.08	Rest
Min			21	4.5	2.5	0.02	

Table 2 – Sample parameters at the various stages of deformation.

Number of passes	Initial diameter [mm]	Final diameter [mm]	Total strain [ε]
1	20.2	14.0	0.74
2	14.0	9.9	1.42
3	9.9	6.9	2.15
4	6.9	5.1	2.75
5	5.1	4.1	3.17
6	4.1	3.5	3.52
7	3.5	3.3	3.64
8	3.3	3.0	3.84

The samples were observed along the directions parallel and perpendicular to the direction of extrusion. The microscopic observations were carried out using a JEOL JEM 1200 EX II with an accelerating voltage of 120 kV. The mean grain size d_{eq} (defined as the diameter of a circle which has the surface area equal to the surface area of a given grain) was determined using a computer equipped with an image analyzer. The value of d_{eq} was determined in more than 200 randomly selected grains.

The existing phases were identified and their volumes estimated by an X-ray diffraction (XRD) analysis, performed at room temperature using a Bruker diffractometer D8 Advance, working with Cu K_α radiation ($\lambda_{K\alpha}$ = 0.154 nm). The authors used Averbach and Cohen method [31] according to ASTM standards [32]. The integrated intensities of the austenite (200) and (220) and the ferrite (200) and (211) diffraction peaks are measured providing four austenite/ferrite peak intensity ratios. The use of multiple diffraction peaks minimizes the effects of preferred orientation providing better measurement accuracy.

The influence of the deformation degree on the mechanical properties of the material were examined by tensile tests carried out in an MTS Q/Test 10 test machine at room temperature with a uniaxial quasistatic strain rate of 10^{-3} s^{-1}. In each deformation state three tensile tests were carried out.

After failure, the tensile specimens were examined in a Hitachi 3500N scanning electron microscope (SEM).

3. Results

3.1. Optical Microscopy

Figs. 1 to 3 show the microstructure of the material before and after the HE process. The elongated austenite grains (white), visible in Fig. 1b, are typical of an extrusion process. HE resulted in the intense refinement and elongation of grains along the extrusion axis. As the deformation along the direction transverse to the extrusion axis increased, the shape of the grains did not change but the microstructure underwent further fragmentation (Figs. 2 and 3). As a result of HE phases are not identifiable by means of optical microscopy. For further microstructural characterization of the tested material TEM will be used.