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Comparison of Grain Refinement in Selected Materials Subjected to Hydrostatic Extrusion

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The subject of this study was to examine and compare the impact of intense hydrostatic extrusion on grain refinement in three different alloys: duplex stainless steel, commercially used aluminum alloy (6060) and Ag-Cu12 alloy. As a result of the process grain sizes from 370 nm to 90 nm were obtained in aluminum and duplex steel. To analyze the of hydrostatic on mechanical properties tensile tests were also carried out. The highest grain refinement (70 nm) and yield strength increase (over 300%) was observed in duplex steel after hydrostatic extrusion.

Keywords: Hydrostatic Extrusion, Severe Plastic Deformation, Nanomaterials, Duplex Steel, Aluminium Alloy, Silver Alloy.

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1. INTRODUCTION

Nanomaterialsare defined as substances where at least one dimension is less than approximately 100 nanometers. This has a significant impact on the observed properties comparing to the commonly used materials [1,2]. The scale effect causes increased ratio of surface area to volume and excess energy in the material. There are two main strategies to achieve this. In the first one where the material is formed atom by atom is called bottom-up approach. The second group involves the transformation of the initial structure to nanoscale material and is referred a stop–down. So far this is the most effective way to obtain bulk nanomaterials.

Severe plastic deformation (SPD) belongs to the top-down approach and is a generic term describing a group of methods used to produce ultrafine-grained and nano-materials [3]. The concept of this method is to induce large strains during forming. To achieve this special tool geometries are used to prevent the free flow of material. Due to large deformation the microstructure undergoes a series of transformation. During the process high densities of crystal lattice defects are produced, particularly dislocations, this results in a significant refining of the grains. The most common methods in this group are equal channel pressing (ECAP)[4,5], high-pressure torsion (HPT) [6,7] and hydrostatic extrusion (HE) [8,9]. After the process the material exhibits increase in strength and reduced elongation caused by decrease in dislocation mobility. Various materials can react differently to large deformations. In particular, the grain refinement process is still not fully understood by the researchers [10].

Hydrostatic extrusion is a process in which the material is pushed through a die of the desired cross-section. The process is carried out in a sealed cylinder in a hydrostatic medium in which the piston movement produces pressure to extrude the rod. The schematic of the process is shown in the Fig. 1. During the process large grain refinement and high strains are achieved under subsequent extrusion processes[11]. What is noteworthy the material is continuously cooled which greatlyreducesgrain growth that may occurdurin deformation. Recent developments in hydrostatic extrusion lead to significant grain refinement, down to nanometric scale for different materials like aluminum [12], titanium [13], and austenitic steel[14].

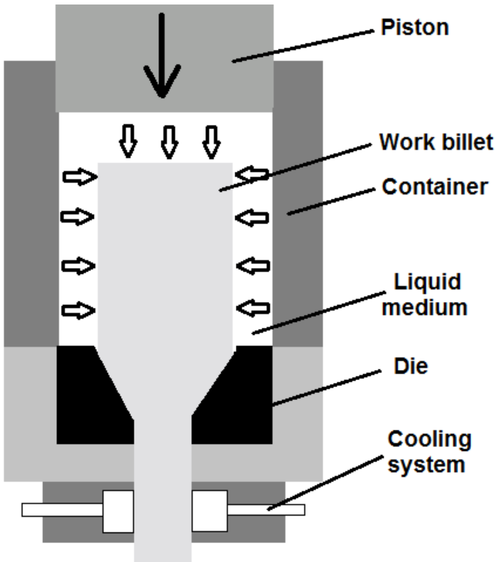

Fig. 1 - Schematic representation of the hydrostatic extrusion process

2. EXPERIMENTAL PROCEDURE

The materials used in the investigations were a commercial 1.4462 duplex stainless steel, Al-Mg-Si (6060) aluminum alloy and Ag-12Cu alloy. The three materials after hydrostatic extrusion were deformed to approximately ε=3.5. The processes was performed at the Institute of High Pressure Physics "UNIPRESS" (Poland).

Transmission Electron Microscopy (TEM) was used to study the microstructural evolution. Observations were carried out on section perpendicular to the extru-

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sion direction. The mean grain size (deq defined asthe diameter of a circle which has the surface area equal to the surface area of a given grain) was determined by a computer equipped with an image analyzer. The deq value was determined for more than 200randomly selected grains. The microscopic observations were carried out using a JEOL JEM 1200 EX II with an accelerating voltage of 120 kV.

The tensile tests were conducted on MTS Q/Test 10 machine with a uniaxial quasistatic strain rate of 10-3 s-1.

The aim of this research was to compare microstructure and mechanical properties of three different face-centred cubic (FCC) metals.

3. RESULTS

Fig. 2. shows the microstructure of Ag-12Cu alloy subjected to hydrostatic extrusion. HE process resulted in fragmentation of the microstructure, and consequently the average output grain size equal to 90 nm was obtained. It should be noted that the deformation led to the apparent fragmentation of original grains and their elongation. There were also regions with high dislocation density in the vicinity of grains free of dislocations. Electron microscopy observations revealed the appearance of plastic deformation bands which contain grains with the smallest size. The selected area diffraction (SAD) patterns indicate that the microstructure contains grains separated by high angle grain boundaries.

In contrast the structure of duplex stainless steel after hydrostatic extrusion is more diverse (Fig. 3). Ferrite forms ultrafine equiaxed grains with diameter of approximately 0.37 μm. There are still some dislocation walls and clusters that didn't yet evolve to subgrains. The austenite regions are much more fragmented. Nanosized blocks of diameter about 70 nm circumnavigate along elongated bands through the material. The SAD-patterns are blurred and stretched (especially for austenite) compared to other materials. This indicates a high disorder in the crystallographic structure and very small grain size with large misorientation.

Completely different from the previously described material is the microstructure of aluminum alloy (Fig. 4). Hydrostatic extrusion to the value of $\varepsilon = 3.5$ leads to the refinement on microstructure and obtain the average grain size $d_{eq} = 0.35$μm. The SAD pattern from this section consists of diffuse rings characteristic of polycrystalline materials. These spots form nearly continuous diffraction rings, which suggest a large number of high angle grain boundaries. The observation indicates that most of the grains are free of dislocations or separated into cells. This suggests that the microstructure of investigated alloy is related to dynamic recovery under large strain. Dynamic recovery occurs easily during aluminum deformation at room temperature due to the high value of stack fault energy. (SFE) The severe strain induced in the material provides enough energy for the dislocations to move from the interior of the grains to the vicinity of the grain boundaries. However, ome areas with high density of dislocations are visible.

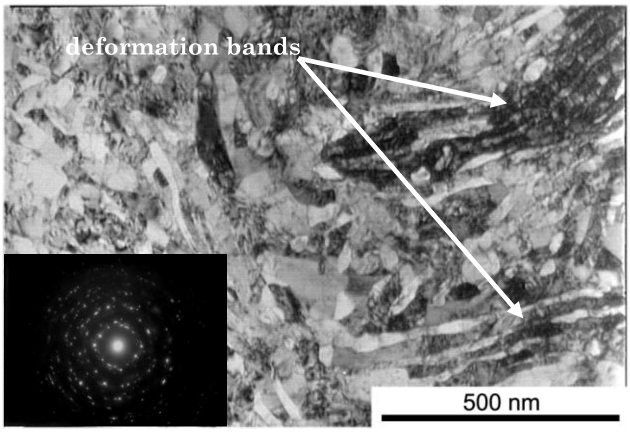

Fig. 2 - Bright field TEM images of Ag-12Cu alloy processed by HE (insets are the corresponding selected-area diffraction patterns).

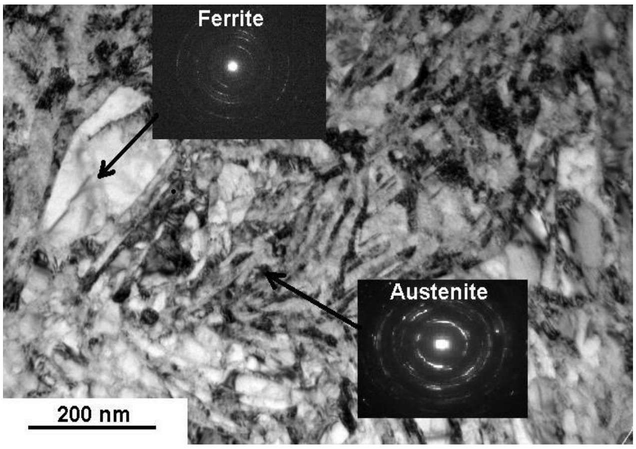

Fig. 3 - Bright field TEM images of the duplex stainless steel processed by HE (insets are the corresponding selected-area diffraction patterns).

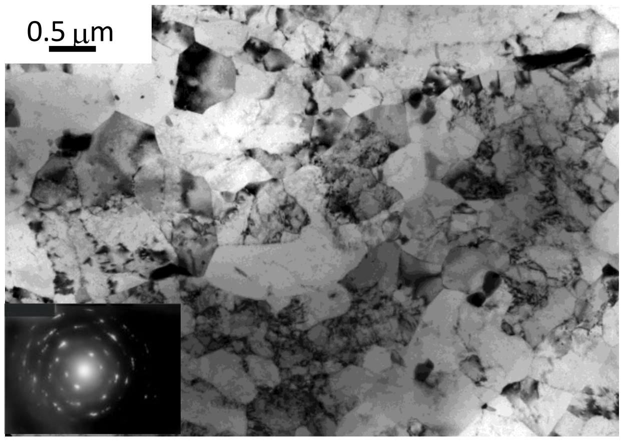

Fig. 4 - Bright field TEM images of the aluminum alloy processed by HE (insets are the corresponding selected-area diffraction patterns).

For mechanical properties tensile tests were conducted for each material before and after hydrostatic extrusion. The results are summarized in Table 1. According to assumptions strength of the materials rose and maximum elongation decreased after hydrostatic extrusion in all materials.

The largest force was needed to deform duplex stainless steel although the maximum elongation was the lowest. What is worth noting the percent change of strength -yield stress (YS) 309%, ultimate tensile strength (UTS) 250% and maximum elongation (10%) after hydrostatic extrusion were the most significant from all tested materials. In case of the aluminum the mechanical properties altered only slightly compared to the initial state. Yield stress increased about 170 % and ultimate tensile stress rose to 131% a decrease in the maximum elongation was insignificant compared to initial state. What is worth noting the total strain for aluminum is merely ε=3.5 whereas some researchers achieved ε=60 using cyclic extrusion-compression[15]. The change of properties for Ag-12Cu alloy was moderate compared to previously described materials. The highest increase of strength was accompanied by the largest decrease in grain size which is in accordance with the Hall-Petch equation.

Table 1 - Average grain size and mechanical properties of tested materials

Material	Yield stress [MPa]			Ultimate tensile strength [MPa]			ε_{MAX}[%]			d_{eq}[μm]	
	Before HE	After HE	Change ratio [%]	Before HE	After HE	Change ratio [%]	Before HE	After HE	Change ratio [%]	Before HE	After HE
Duplex steel	572	1770	309%	748	1872	250%	23.4	2.4	10%	Ferrite 4.3 Austenite 3.7	Ferrite 0.37 Austenite 0.07
Ag-12Cu	378	630	167%	382	800	209%	13.5	5.6	41%	0.46	0.09
Aluminum 6060	170	290	171%	261	341	131%	16.2	13.4	83%	1.24	0.35

4. DISCUSION

The aim of this study was to evaluate microstructure and mechanical properties changes for different material subjected to hydrostatic extrusion. To achieve this three different metals with FCC crystallographic structure were extruded to a total strain of 3.5. TEM was carried out to compare the microstructure changes which have taken place under the influence of plastic deformation. Additional tensile tests were conducted in order to compare the mechanical properties of tested materials.

The largest grain refinement was observed for duplex stainless steel along with it the highest strength needed to deform the material. This is directly related to the increase of dislocations density and grain refinement. The dominating mechanism of grain refinement all materials was subdivision by dislocation walls. During deformation density of dislocations increases rapidly. Cellular structures with incidental dislocation boundaries are formed within the grains and cell blocks separated by geometrically necessary boundaries. With further increase of strain misorientation grows and subgrains are created [4].The process is continuously repeated leading to structure fragmentation although there is a certain asymptotic limit that the grain size tends resulting from thermodynamic conditions. Additional mechanisms that facilitate dislocation accommodation increase grain refinement [16]. An example of such behavior was observed in austenite of duplex stainless steel where twinning occurred. The structure was most fragmented from all tested materials.

The two primary methods of deformation in metals are slip and twinning. Slip occurs by dislocation glide of either screw or edge dislocations within a slip plane. Slip is by far the most common mechanism. Twinning is less common but readily occurs under some circumstances [17]. Twinning proceeds when there are not enough slip systems to accommodate deformation and/or when the material has a very low SFE. Twins are abundant in many low SFE metals like copper[18], nickel [19]and silver [20].The microstructure observations in deformed, low SFE FCC materials (duplex steel and silver alloy) are often difficult to characterize because of the complexity of the deformation microstructures; the latter usually result from a combination of dislocation glide and fine mechanical twinning and then, at higher strains, shear banding at different scales.

It has been proved that SFE is an important material parameter that affects the deformation mechanisms and the mechanical behavior of metals and alloys[21].In the case of aluminum alloy which is a material with high SFE (~ 160mJ/m^2)plastic deformation process is realized by slip. Additionally during deformation occurs dynamic recovery. The severe strain induced in the material provides enough energy for the dislocations to move from the interior of the grains to the vicinity of the grain boundaries. In the case of duplex steel and Ag-Cu alloy the deformation and grain refinement were facilitated. The structures of low SFE material tend to twin and create partial rather than screw dislocation. Due to this five or more slip systems must be active for large deformations. When shear deformation and twinning occurs at the same time grains align towards a more preferred orientation such as <111> and <100>. This leads to formation of bands both seen in duplex stainless steel and Ag-Cu alloy[22].

The observed forces needed for deformation were higher than in the initial state for all materials after hydrostatic extrusion. This is due to the increase of defects concentration and grain refinement in the material. However the increase of strength is at the expense of elongation. This is particularly notice able for the duplex steel where the most significant changes were observed. The SAD patterns were most irregular and blurred for austenite of the studied materials. For the rest of the metals the changes of microstructure weren't so noticeable and thus the mechanical properties differed not so much from the initial state.

5. CONCLUSIONS

From all tested materials subjected to hydrostatic extrusion (strain ε=3.5)only two with the lowest SFE exhibited nanocrystalline structure – duplex steel and Ag-12Cu alloy. In each case the main mechanism was shear deformation and twinning.

The largest increase in strength of the material was observed for duplex stainless (increase over 300% in UTS) steel and the smallest for aluminum 6060 (130% increase in UTS). This is directly correlated to the deformation mechanisms in both materials. Additional twinning and large grain refinement in the austenite phase resulted in more effective blockade of dislocation movement.

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