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## SEVERE PLASTIC DEFORMATION BY MULTISTAGE COMPRESSION

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### ABSTRACT

This paper covers the problematic of creating ultra-fine grained metals by severe plastic deformation methods. Unlike surface treatment technologies which create thin layers of nanomaterials, severe plastic deformation methods can create nanosized grains almost within whole volume by introducing high values of effective strain. In this paper, compression by V-shape dies has been presented as method for creating ultra-fine grained metals. Friction coefficient between the V-shape dies and sample has been varied in order to find its influence on effective strain values at the center of specimens. Grain size at the sample has been analysed by using transmission electron microscope.

**Key words:** severe plastic deformation, compression by V-shape dies, ultra-fine grain, effective strain, numerical simulation, transmission electron microscopy

### 1. INTRODUCTION

In recent years, a number of methods for refining the structure of metals by severe plastic deformation (SPD) have been developed. Some of those methods permit grain refinement to a nanometric level. Numerous investigations show that the metals having such a structure are characterized by a number of specific properties including significantly higher yield point than that produced by conventional deformation methods (rolling, drawing) [1].

The most important concern is to combine manufacturing process with metal fabrication. The selfassembly, non-traditional lithography, templated growth and biomimetics are some of the potential technologies [2]. In such processes, nanoparticles, delivered in the form of nanotubes, nanopowders, quantum-dots, and biomaterials are staked into final product in a designed way. The most challenging problem is the cost of making many of the raw components for functional

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nanomaterials. The cost of producing these materials often exceeds \$700/g and makes potential products economically infeasible. The time required for performing any engineering work at nano scale is also considerable for methods based on the material synthesis.

Another major challenge is the lack of chemical, morphological, or mechanical stability in many nanomaterials. These often lead to spatial distortions, suboptimal thermal behavior, reduced mechanical response and poor electrical properties, which lower the overall system performance. An alternative approach is to employ bulk nanomaterials and traditional shaping methods while constructing systems and devices at the micro and nano scales. However, conventional metallurgy cannot supply metals featuring grains substantially smaller than characteristic dimensions of the engineering micro-components. This and the encouraging economic forecasts for nanometals cause great interest in the development of new fabrication techniques, with mass production scale-up capabilities and low-cost [3].

The technologies claiming bulk capability include electrodeposition and crystallization of initially amorphous metals. However, the two main competing technologies are compaction and sintering of nanopowders and severe plastic deformation of bulk metals. The latter process avoids the presence of impurities or porosity typical of powder metallurgy. It involves generation of a very large plastic strain in coarse-grain bulk metals using one of the newly developed metal forming processes [4, 5].

It is worth emphasizing that all metallic materials respond to severe straining in basically the same way. Technically, nanostructure can be achieved locally by a number of processes like cold rolling [6] or friction wear. However, these processes are difficult to control in terms of grain refinement in the whole deformed part and there is no room for the manipulation of grain structure efficiently [7].

## 2. GRAIN REFINEMENT

A simultaneous accumulation of localized dislocations and increase in the lattice misorientation are responsible for crystal subdivision and subsequently developed submicroscopic grains. However, some researches reveal that, particularly in pure metals, there exists a limit below which reducing the grain size further results in shifting in the deformation mechanism into a different yet unknown mechanism of plastic flow [3].

Under an applied stress, existing dislocations and dislocations generated by Frank–Read Sources will move through a crystalline lattice until encountering a grain boundary, where the large atomic mismatch between different grains creates a repulsive stress field to oppose continued dislocation motion. As more dislocations propagate to this boundary, dislocation 'pile up' occurs as a cluster of dislocations are unable to move past the boundary. As dislocations generate repulsive stress fields, each successive dislocation will apply a repulsive force to the dislocation incident with the grain boundary. These repulsive forces act as a driving force to reduce the energetic barrier for diffusion across the boundary, such that additional pile up causes dislocation diffusion across the grain boundary, allowing further deformation in the material. Decreasing grain size decreases the amount of possible pile up at the boundary. Therefore, the higher the applied stress for dislocation movement the higher the yield strength is (Fig. 1). Thus, there is then an inverse relationship between grain size and yield strength, as demonstrated by the Hall–Petch equation (Fig 2) [9].



Fig.1 - Grain size and dislocation pileup [8]

Obviously, there is a limit to this mode of strengthening, as infinitely strong materials do not exist. Grain sizes can range from about 100  $\mu$ m (large grains) to 1  $\mu$ m (small grains). Lower than this, the size of dislocations begins to approach the size of the grains. At a grain size of about 10 nm, [11] only one or two dislocations can fit inside of a grain (Fig. 1). This scheme prohibits dislocation pile-up and instead results in grain boundary diffusion. The lattice resolves the applied stress by grain boundary sliding, resulting in a decrease in the material's yield strength (Fig. 2). Alloys are more responsive to intensive straining than pure metals, which results in finer grains [12].



Fig.2 - Graphical illustration of the theoretical limit for grain boundary strengthening [10]

Valiev [13] formulated three requirements to obtain materials with submicron grain size: the fine grained material must have predominantly high angle boundaries, the structure must be uniform over the sample volume and the large plastic strains may not have generated internal damage or cracks. Traditional deformation methods like rolling and wire drawing cannot meet these requirements. Therefore special deformation methods have been developed.

#### **3. SPD METHODS**

Ultra-fine grained material was accomplished in a controllable way, was due to an equal channel angular extrusion (ECAE) process introduced by Segal in the 1970s [7]. ECAE, also known as ECAP, gave an impetus to the development of the whole range of similar processes which led to establishing a new discipline of metal forming – SPD methods.

A billet of the test material is pressed through a die consisting of two channels with identical cross sections, intersecting at an angle  $\Phi$ , usually  $60^{\circ} < \Phi < 135^{\circ}$  and often  $\Phi = 90^{\circ}$  (Fig. 3). Some dies have a rounded corner with angle  $\psi$ , others have  $\psi = 0$ . The deformation occurs by simple shear parallel to the intersecting plane of the channels [14].

In spite of the actual popularity of the technique, some drawbacks of ECAP must be recognized. ECAP is a discontinuous process with limitations in up-scaling potential. Moreover, the volume fraction of useful material (with uniform microstructure and without cracks) can be rather low because only the portion of the billet that has passed through the shear zone, will receive the desired deformation and grain refinement. Barber et. al. figured out that for a sample with square cross section and an aspect ratio of 6, after 8 ECAP passes route A, only ~30% of the material is fully worked as intended. For route B, the efficiency is ~45% and for route C it is ~83% [15].



Fig.3 - Schematic illustration of a ECAP SPD method [14]

Other significant continuous SPD methods which increase the efficiency of ECAP are developed. They combine the concept of ECAP with classical rolling and include asymmetric rolling, continuous repetitive corrugation and straightening and conshearing process (Fig. 4).



Fig.4 - Continuous SPD methods a) asymmetric rolling, b) continuous repetitive corrugation and straightening, c) conshearing process [14]

Another significant SPD technique is high-pressure torsion (HPT). Small discs (typically 10-20 mm diameter and 0.2-1 mm thick) are strained in torsion under an applied pressure of several GPa (Fig. 5). Although in a classical torsion test it is generally assumed that the center of the sample is



material

Fig.5 - Schematic illustration of a high-pressure torsion SPD method [14]

## 4. SPD BY V-SHAPE DIE COMPRESSION

Discontinuous SPD method for upsetting square shaped billet by V-shaped dies is presented on Fig. 6 while V-shape die and billet geometry is presented on Fig. 10.



Fig.6 - V-shape die compression SPD method

V-shape die compression is multi-stage process in which, after single compression stage is completed, sample is removed from the dies and rotated for 90° in counter-clockwise direction and returned in the dies. Compression by V-shape die is completed when crack occurs inside of sample or when stroke in single compression stage becomes too low.

During multi-stage compression by V-shape dies workpiece's cross-section is reduced, since it's length is increasing. By simulating compression process it is found that increase of friction coefficient reduces material flow in longitudinal direction. In this manner reduction of workpiece's cross-section is slowed down and number of forming stages is increased. Compression by V-shape dies has been simulated by Simufact.Forming V10.0.1 software where friction coefficient value has been varied in order to find its influence on effective strain at the end of multi-stage compression.

Figures 7 to 9 displays effective strain distribution in samples with different friction coefficient

values between V-shape dies and samples, and maximum number of compression stages obtained for selected friction coefficient.

Experimental application of compression process by V-shaped dies was conducted with the dies whose dimensions are presented on Fig. 10 and which were made of X210Cr12 cold work tool steel (Č.4150). Billets with dimensions according to Fig. 10 were made of CK15 unalloyed carbon steel (Č.1221). Hardness of the dies was 58+2 HRC and there was no lubrication medium used between dies and samples.



Fig.7 - Effective strain distribution after 5th stage compression by V-shape dies for  $\mu$ =0.12



Fig.8 - Effective strain distribution after 7th stage compression by V-shape dies for  $\mu$ =0.3



Fig.9 - Effective strain distribution after 8th stagecompression by V-shape dies for  $\mu$ =0.4

Sample has been compressed in eighteen phases. Further compression was not possible since compression stroke in last stage was too low to perform compression since sample's cross-section is reduced in previous compression stages. Also, after sample compression for eighteen times, no crack was found in sample's material. By analyzing the microstructure of initial samples it has been found that the average grainsize is 18.9  $\mu$ m.



Fig. 10 - V-shape die and billet geometry

Preparation of the samples for transmission electron microscopy (TEM) analysis has been conducted by focused ion beam (FIB) - Quanta<sup>TM</sup> 3D FEG. TEM sample's dimensions are usually 5x4  $\mu$ m in length and width (Fig. 11a) and between 100 and 200 nm in thickness (Fig.11b).



Fig.11 - a) TEM sample on workpiece compressed eighteen times, b) thickness of the TEM sample

After the samples fabrication the mounting on the sample holder was carried out by robotic arm. Transmission electron microscopy analysis was carried out by JEOL JEM-2010using contrast mode 2 and 3. TEM analysis revealed that the grain size has been successfully reduced below 1  $\mu$ m (Fig. 12) typically ranging from 200 to 700nm.



Fig.12 - Grain size in sample compressed eighteen times

### 5. CONCLUSIONS

It can be concluded that V-shape dies can be successfully used for creating ultra-fine grained material since grain size is reduced below 1  $\mu$ m which has been confirmed by TEM analysis. Proposed die geometry successfully introduces high values of effective strain thus enabling grain subdivision. From the results obtained in simulation it can be seen that increase of friction coefficient value leads to an increase of the number of compression stages and increase of total value of effective stress. Future research will include grain size uniformity, hardness and formability analysis on the samples shaped by V-shaped die compression.

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# INTENZIVNA PLASTIČNA DEFORMACIJA VIŠEFAZNIM SABIJANJEM

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#### REZIME

Ovaj rad prikazuje problematiku stvaranja metalnih materiala sa ultra-finim zrnima metodama intezivne plastične deformacije. Za razliku od tehnologija površinskog inžinjeringa koje formiraju tanke slojeve nanomaterijala, intenzivna plastična deformacija može formira zrna u nanometarskom opsegu skoro po celoj zapremini obratka tako što uvodi visoke vrednosti efektivne deformacije. U ovom radu je prikazano sabijanje pomoću alata V oblika kao efektivni metod za dobijanje metalnih materijala sa ultra-finim zrnima. Koeficijent trenja između alata V oblika i uzoraka je variran kako bi se pronašao njegov uticaj na vrednosti efektivne deformacije u središtu uzorka. Veličina zrna u uzorku je analizirana pomoću transmisionog elektronskog mikroskopa. **Ključne reči:**intenzivna plastična deformacija, sabijanje alatima V oblika, ultra-fina zrna, efektivna deformacija, numerička simulacija, transmisioni elektronski mikroskop