Refining Structure of Copper by SPD process

J Švec¹, P Szkandera¹, S Rusz¹, O Hilšer¹ and J Petrů

¹VSB – Technical University of Ostrava, Faculty of Mechanical Engineering, 17. listopadu 15, 708 33 Ostrava – Poruba

E-mail: stanislav.rusz@vsb.cz

Abstract. This research concerned the whole production of UFG materials, using Severe Plastic Deformation (SPD). These new technologies for production of semi-finished products with ultra-fine grained structure differ from conventional technologies. One of them is new type of equipment DRECE (Dual Rolling Equal Channel Extrusion), designated for obtaining UFG structure and increase of mechanical properties in strip of sheet. The main goal of the paper is a review of current achieved results obtained by processing of Cu based materials by DRECE method, the equipment of which was put in service towards the end of 2009 at the Department of mechanical technology, Faculty of mechanical engineering, VSB - Technical University of Ostrava. Cu-based materials – technical pure Cu (99.3% purity) – is used as experimental material. Experiments with use of the formed structural material - copper was made on the forming device in order to achieve grain refinement in the strip of sheet (Cu 99.3% purity) with dimensions 58 x 2 x 1000 mm. The paper describes results of determination of an influence of number of passes on resulting mechanical and micro-structural properties of the processed Cu-based materials.

Keywords: severe plastic deformation, DRECE method, copper, mechanical properties, heat treatment, structure

Introduction

In many technical processes of forming the deformation is substantially greater than conditions at the tensile test. These new activities demonstrated at the beginning of the nineties that it was possible to manufacture nano-crystalline metallic materials by very high plastic deformation at low homological temperatures. It is possible to achieve in ductile metallic materials at the tensile test a deformation from 30% to 70% [1-2]. Obtaining of nano-crystalline structures requires typical magnitudes of deformation of the order from 100 to 1000%. High deformation at comparatively low homological temperatures is an efficient method for manufacture of ultrafine grained (UFG) massive materials [3]. New technologies, which use high deformation for obtaining the fine-grained structure is described namely by Valiev [4]. This research concerned the whole production of UFG materials, using Severe Plastic Deformation (SPD). Several types of SPD technologies serving for production of UFG metals were developed already at the beginning of the nineties. One of them is new type of equipment called DRECE (Dual Rolls Equal Channel Extrusion), designated for obtaining UFG structure in a strip of sheet.

1 Experimental materials and procedures

A sheet from commercial pure aluminum was used at the very beginning of experimental works [5]. This material was used for practical solving of the first technological problems with jamming and
high warp of the processed sheets. Copper-based materials were selected for next experiments after performed literature search and after obtaining of the binary phase diagrams (in the case of alloys). Chemical composition of copper is shown in Table 1.

Table 1. Chemical composition of copper (wt%)

| Sn  | Al  | As  | Pb  | Sb  | Fe  | Cu  |
|-----|-----|-----|-----|-----|-----|-----|
| 0.15| 0.05| 0.1 | 0.1 | 0.08| 0.05| Rest|

Sheets of dimensions (58x2x1000 mm) were used as an input material. The sheets were bought in commercial trade network in as cold-rolled state, without previous heat treatment, in chemical purity corresponding to the relevant standard. Forming equipment based on process CONFORM, modified for sheet forming. During 2009 a prototype of this equipment was put into trial operation at the working site of the VSB - Technical university of Ostrava [5-6]. Fig. 1 shows a principle and overall view of the prototype of this equipment. It consists of the following main parts: gear of the type Nord with electric drive, disc clutch, feed roller and pressure rollers with regulation of thrust, forming tool made of the steel grade Dievar. Strip of sheet is fed into the working space and it is pushed by the feed roller with help of pressure rollers through the forming tool without change of its cross section. Next pass is realized after rotation of sheet by an angle of 180°. Repeated plastic deformation realised in this manner brings substantial refinement of structure. During the trial operation first experiments were made followed by their evaluation. On the basis of these works some modifications of design were proposed [6].

Both experimental materials were formed by SPD process at ambient temperature without previous heat treatment and selected samples operative heat between individual passes. The sheets were rotated between individual passes by 180° around their longitudinal axis [5]. Forming processed sheets were subjected to metallographic evaluation and basic mechanical properties were determined.

The extruded samples of used materials after completion of selected passes were then cut from sheets into individual series for preparation of individual testing specimens for mechanical tests and metallographic evaluation, which were marked by symbols Cu-X, where X expresses the number of passes (X = 1-6 or 10). The sample of initial state is marked with the symbol 0. Mechanical properties of studied samples were tested by tensile test and by Vickers hardness method. Metallographic analysis of structure was made on light microscope and with use of TEM.

2 Description and discussion of achieved results

Mechanical properties of studied samples were tested by Vickers hardness method on the HPO 250 testing device. Average values of hardness from five measurements were calculated. Tensile test was performed at ambient temperature on the Inova TSM 50 testing machine.
2.1 **Hardness test**

Results of Vickers hardness test for the samples of copper are shown in Table 2.

As it can be seen from Table 2 these values rapidly increased from the 1st to the 4th pass. After the 5th pass these values increased slightly and reached even the value of 119 HV5 in the case of Cu-10.

**Table 2** Average values of hardness of Cu in the samples

| Number of passes | Hardness HV5 |
|------------------|--------------|
| Cu-0             | 84           |
| Cu-2             | 98           |
| Cu-4             | 101          |
| Cu-6             | 108          |
| Cu-8             | 109          |
| Cu-10            | 119          |

As it can be seen from Table 2 for samples without heat treatment, these values rapidly increase from the 1st to the 4th pass. After the 4th pass the value of hardness stays nearly the same. It may be assumed from this dependence, that the biggest increase of hardness caused by dislocation strengthening in the course of plastic deformation occurs till the 4th pass and subsequent passes do not contribute substantially to further increase of strengthening. Values of hardness are in the cases of the 2nd and the 4th pass slightly lower than those of initial state, and in the case of the 6th pass they are higher than in initial state. These values of hardness are also increasing with an increasing number of passes.

2.2 **Tensile tests**

Tensile test of the samples was made on the testing machine Inova TSM 50 at the strain rate $1 \times 10^4 \text{s}^{-1}$. Results of tensile test of the copper samples are given in Table 3 and they are shown in Figure 2.

**Table 3** Influence of SPD process on mechanical properties

| Number of passes | $R_{p0.2}$ [MPa] | $R_m$ [MPa] | $A_{80}$ [%] |
|------------------|-------------------|-------------|--------------|
| Cu-0             | 198               | 264         | 37.3         |
| Cu-2             | 252               | 284         | 12.6         |
| Cu-4             | 255               | 311         | 8.7          |
| Cu-6             | 247               | 324         | 6.8          |
| Cu-8             | 231               | 308         | 6.4          |
| Cu-10            | 223               | 315         | 4.4          |
It is obvious from the results of tensile test on copper samples that the biggest increase of yield strength occurs after the 2nd pass and that during the next passes it gradually slowly decreases, while the strength limit gradually increases till the 6th pass and it stagnates during subsequent passes. These results are reflected in considerable drop of ductility after the 2nd pass to the value of 12.6%. During next passes it decreases slowly to the value of 4.4% after the 10th pass. These results correspond with dependence of the number of passes on the hardness values given in the previous chapter. Results of tensile test of samples Cu (99.3 % purity) without heat treatment are given in Tab. 3 and they are shown in Figure 2. As it can be seen from this figure the yield stress and ultimate tensile stress after DRECE processing are increased till the 4th pass, while the elongation is decreased. After the 6th pass, similarly as in the case of copper, these values slightly decrease. These results are also in agreement with determination of hardness values given in the previous chapter.

2.3 Metallographic analysis

For investigation of structure a metallographic analysis was made on light microscope NEOPHOT 2. Initial state was compared with the state after selected number of passes. More precise analysis was made on TEM microscope. After usual metallographic preparation the samples were electrolytically etched. Figures 3 and 4 show the microstructure of selected samples of copper. Microstructure of initial state of the Cu samples is shown in Figure 3a. This microstructure consists of grains in agreement with the fact that this material was formed before the forming process. Grain size reached the value G4 according to the ASTM. Microstructure of the sample after the 4th, 8th and 10th pass through the DRECE tool is shown in Figure 3 b, c, d. Refining of grains after each pass was only small as it can be seen in Figure 3d showing the result after last passes. Grain size decreased on the value G5-G6 according to the ASTM. More information by TEM analysis was acquired as it can be seen in Figure 4. This picture shows increasing dislocations’ density and presence of slip band. This fact is in agreement with the hardness measurement. In further work, for the exact analysis of the structure, it is necessary to analyze a larger number of samples on TEM and SEM.
### Figure 3
Structure of pure Cu: Initial state (a) and after 10 DRECE passes (b)

| a) | b) |
|----|----|

### Figure 4
TEM images of investigated material: initial state (a); after 4 passes (b); after 8 passes (c) and after 10 passes (d)

| a) | b) | c) | d) |
|----|----|----|----|

---

#### 3 Summary

The device has been designed as a prototype equipment for production of UFG structure in a strip of sheet made of non-ferrous metals with subsequent possibility of deformation, and also in a strip of steel sheets with thickness of 2 mm. Creation of UFG structure in the strip of sheet is closely connected to the design of suitable geometry of the forming tool,
appropriately dimensioned power unit and control system enabling setting of various values of peripheral velocities. It is also necessary to optimise the pressure force and thus also pressure on the pressure rollers, so that only extrusion occurs, but not the rolling. From the viewpoint of the forming parameters higher number of passes will bring considerable strengthening of the formed material. The forming equipment is at the stage of verification and future works will verify the influence of technological parameters on the increase of efficiency of SPD process for obtaining the UFG structure in non-ferrous metals. According to the level of the obtained results of extrusion of examined materials it is possible to state that the equipment is fully functional. It may be assumed from dependence of mechanical properties on the number of passes, that the biggest increase of hardness caused by dislocation strengthening in the course of plastic deformation occurs till the 4th pass and subsequent passes do not contribute substantially to further increase of strengthening. The results show the necessity of next experiments especially in terms of for the analysis the changing of electrical parameters (electro-conductivity).

Acknowledgments
The authors would like to acknowledge gratefully the Ministry of Education, Youth and sports of Czech Republic for its support to the project “Creation of an international team of scientist and participation in scientific networks in the sphere of nanotechnology and unconventional forming metal”, CZ.1.07/2.3.00/20.0038, the project Pre-seed Materials IA3, Production technology of a sheet strip with an ultra-fine grain structure, No. CZ.1.05/3.1.00/14.0320 and project FRVS, IRP No. 239/18 Innovation of teaching subject - Forming

References
[1] GUTKIN, M.YU., OVIDKO, I.A. AND PANDE, C.S. Theoretical models of plastic deformation processes in nanocrystalline materials, Reviews on Advanced Materials Science, September 2001, vol. 2, p. 80 – 102.
[2] KOBAYASHI, M. et al. Research and Development of Superplastic Materials „Recent Progresses and Future Prospects“, Metallurgical Transactions 18A, 1987, p. 685-695.
[3] RUSZ, S., ČIŽEK, L., MICHENKA, V., DUTKIEWICZ, J., SALAJKA, M., HILŠER, O., TYLŠAR, S., KEDROŇ, J., KLOS, M.: New type of device for achievement of grain refinement in metal strip. Advanced Materials Research. Vol. 1127 (2015), Switzerland: Trans Tech Publications, p. 91 - 97.
[4] VALIEV, R. Recent developments of severe plastic deformations techniques for processing bulk nanostructured materials, Materials Science Forum A 579 (2008) p. 1–14.
[5] RUSZ, S., KLYSZEWSKI, A., SALAJKA, M., HILŠER, O., ČIŽEK, L. and KLOS, M. Possibilities of application methods DRECE in forming of non-ferrous metals, Archives of Metallurgy and Materials, Vol. 60, 2015, ISSUE 4, p. 3011-3015, DOI: 10.1515/amm-2015-0481
[6] HILŠER, O., SALAJKA, M. and RUSZ, S. Study of the mechanical properties of steel and selected types of non-ferrous alloys after application of the DRECE process. In NANOCON 2015: 7th International Conference on Nanomaterials-Research & Application. Ostrava: Tanger, 2015, p. 163-167.