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HCV DEFORMATION – METHOD TO STUDY THE VISCOPLASTIC BEHAVIOR OF NANOCRYSTALLINE METALLIC MATERIALS

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Abstract. In this paper, the nanocrystalline structure of pure copper was processed by severe plastic deformation (SPD). The tests were carried out on Materials Testing System INSTRON 8516 in three strain controlled regimes by strain amplitude of 1-2% and by the number of cycles up to 30. The main objective of this study is to elaborate a new (non-destructive) testing method – Hard Cyclic Viscoplastic (HCV) deformation to study of the viscoplastic behavior of nanocrystalline (NC) metallic materials. This testing method can be used to investigate the change of mechanical and physical properties, as well as the changes in the micro- and nano structure, the formation of micro-defects and cracks during tension-compression loading in the viscoplastic state of metals. This testing method can be used also to study the stability and viability of the properties of NC metallic materials.

1. INTRODUCTION

It is well known that the severe plastic deformed (SPD) bulk NC metals exhibit extraordinary high strength and hardness [1,2]. The ultimate tensile strength R_m of NC metals can be 4-5 times higher, Vicker's hardness and microhardness 2-4 times higher compared to the conventional coarse-grained (CG) metals. Unfortunately, these outstanding mechanical properties of NC metals are not stable during the exploitation as the NC metals show low resistance to creep and high stress relaxation rate [3,4] and low viability also [5]. Such materials can withstand the conditions of hard cyclic loading only during very short exploitation time at the relative low temperatures.

Through out the history, a large variety of mechanical testing methods have been developed. The experimental investigation of the mechanical and physical properties behavior of NC metals have been limited in scope and quantity. The material behavior is different under tensile and compressive loads in plastic state. The ultimate tensile strength can be used for purposes of specification and for quality control of a product. For viability assessment of new nanocrystalline metals for a given application, the balance between its properties is essential. For new nanocrystalline materials, the viability assessment can be divided in three segments [6]: viability, market forecasting, and value capture. The viability assessment is an application if the balance between its technical performance, cost of production and value in market is seeked for. A measure of yielding at which plastic deformation is observed to begin depends on the sensitivity of the strain measurements. There are three criteria: the elastic limit, the

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proportional limit, and the yield strength. The measures of ductility and modulus of elasticity can be determined during uniaxial tension test of metals. The ductility indicates the ability the metals deformation without fracture, the behavior in which of metals flow plastically before fracture according to processing or structure state. The resilience and toughness shows the materials ability to absorb energy in the plastic range, for example, during cyclic deformation. In some publications [7-10], NC metals were tested by cyclic deformation method in both stress- and strain-controlled regimes. The low-cycle fatigue (LCF) and high-cycle fatigue (HCF) tests were preformed. The main test parameters are strain amplitude and number of cycles to total fracture. Depending on frequency, the strain amplitudes in percents were between $5\cdot10^{-4}$ to $2\cdot10^{-1}$. It was shown [11,12] that the nanocrystalline copper exhibits the best stability and highest true fracture stress after low temperature heat treatment. The influence of test temperature on strain rate and strain rate sensitivity, and also dependence of strain rate and temperature at flow stress during testing are very important parameters. It can be witnessed from the microstructure of the metallic materials [13].

Depending on material application area the test methods and test parameters can be different. For example, various materials for the aerospace applications are Ti- and Ni-based heatproof microcrystalline alloys, and single-crystal superalloys [14]. These materials are tested at elevated temperatures by HCF method.

In these tests the specimens are subjected to the gradually increasing load cycles until the fracture. These materials are tested at cyclic loads with gradually increasing tensile load. For example, the materials used for compressor and turbine blades for turbo-jet are tested at elevated temperatures of $2\cdot10^{7}$ cycles at frequency of 1000 – 1200 Hz in several testing sequences. If the specimen does not fail after the first sequence (normally $2\cdot10^{7}$ cycles) the test will be continued at higher load levels. The above-mentioned testing method is not suitable for testing of NC metals as these materials exhibit relatively low thermal stability [11,12].

For cyclic deformation in viscoplastic state [11,15-18], the strain amplitudes were increased up to 1-2%. As the strain amplitude is large, the number of cycles to failure is relatively small and also low testing frequencies can be used. Important properties of metals such as transition behavior from strain strengthening to strain softening and its mechanism [18-20], and fatigue life of NC materials [21-23] can be studied by HCV test. As the thermal stability of the NC metals is relatively low they may be used in heavy loaded and light-weight applications for a short term. For example, in aerospace, in rocketry or in military equipments, in which there are the material requirements such as ability to withstand repeated alternative stresses will posses a minimal margin of safety and a minimal relative density by maximal specific strength. The knowledge and understanding of NC metals behavior under mentioned hard cyclic-working condition, and especially by overloading with peak deformation amplitude of 2% by low cycles number and low strain rate, are of great importance. The aim of the present study is to develop a simple and timesaving testing method, so-called hard cyclic viscoplastic (HCV) deformation.

2. EXPERIMENTAL PROCEDURES

Commercial purity copper of cold-drawn state of the following chemical composition: 0.0238Fe, 0.0084Al, and balance-Cu (wt.%) was used as initial material in the present experimental work. This material with rod dimensions of 16 μm in diameter and in 150 mm in length was heat treated at temperature 650 °C during 1.5 h and cooled with furnace. After homogeneous nucleation the mean grain size of copper was increased to 250 μm. The nanocrystalline structure in the material was fabricated by severe plastic deformation (SPD) technique by ECAP process. Eleven pressing through intersecting at 90° round of 16 mm diameter channels was performed at room temperature with 5 $\mathrm{mm\cdot s^{-1}}$ pressing velocity. The 'route B_c' was used; the sample was rotated through 90° counter-clockwise along longitudinal axes (of specimen) between subsequent passes. During each pass of ECAP the sample temperature was increased up to 70-80 °C due to the SPD. The samples after SPD at eleven passes of ECAP were used for HCV deformation without subsequent heat treatment (Sample N1). After SPD the six samples were heat treated up to 200 °C with heating rate at 1 $\mathrm{°C\cdot min^{-1}}$ (Sample N2) and three samples (from these six samples) were double heat treated up to 400 °C with heating rate at 2 $\mathrm{°C\cdot min^{-1}}$ (Sample N3). As a reference the cold-drawn (Sample N4) and the annealed at 650 °C for 1.5 h (Sample N5) both of coarse-grained (CG) structure state samples were used.

The samples for testing with HCV deformation method were manufactured by turning. The geometry of the test region was with dimensions of 10±0.05 mm in diameter and of 30±1 mm in length with radius of curvature of the neck of 3 mm. This region was mechanically polished to remove a damaged layer from the surface.

The HCV deformation multiple test experiments were carried out in three strain controlled regimes: 1 – at 1% of strain amplitude; 2 – HCV deformed specimens at 1% of strain amplitude were subjected to a HCV deformation at 2% strain amplitude; and 3 – the specimens deformed in first two series were reloaded to a strain with amplitude at 1%. In each three, regimes the tests were performed at 30 cycles during 600 s (10 min.). All tests were performed in ambient environmental condition using materials testing system INSTRON 8516. An extensometer with base length of 25 mm was applied on the specimen for exact strain measurement. For the better understanding of the mechanisms of structural changes and to determine the mechanical properties of the tested materials during HCV deformation some of the specimens were subjected to standard quasi-stationary tensile test after HCV deformation. For comparison a tensile test with SPD NC (N6) and annealed CG (N7) copper specimens (without HCV) was performed. The microstructures of specimens before HCV and after HCV in necking region were investigated on SEM Gemini LEO Supra-35. The hardness, mechanical and physical properties of metals change during processing and testing were measured on the universal hardness tester Zwick Z2.5/TS1S. The crystallites mean size was measured on X-ray diffractometer D5005 Bruker AXS and was computed with the WIN-CRYSIZE program.

3. RESULTS AND DISCUSSION

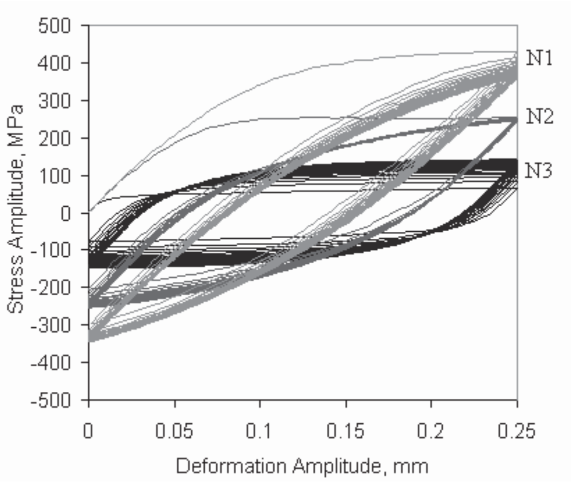

Fig. 1. Hysteresis loops for specimens of SPD NC copper (N1), heat treated NC copper (N2) and double heat treated UFG copper (N3).

The HCV deformation hysteresis loops for first regime (for strain amplitude at 1% for 30 cycles) of the copper with different structures: sample N1, sample N2, and sample N3 are shown on Fig. 1. The NC copper (N1) shows the maximal values and nonsymmetrical stress amplitude during the very first cycles (up to 10) the ultimate stress decreases during tension cycle and increases during compression cycle. The heat treatment at low temperature (N2) influences the tension stress stability. During compression cycle the strain hardening takes place and the ultimate compression stress increases from 204 MPa to 251 MPa. The double heat-treated specimen (N3) hysteresis loop show the strain hardening behavior during tension as well during compression half cycle. The maximal values of the stress amplitude or stress at tension and stress at compression (for cycle 1 and for cycle 30) for all regimes: I - strain amplitude of 1%, II – strain amplitude of 2% (after 1% of strain amplitude during first regime) and III – strain amplitude at 1% (specimen deformed at two first regimes at 1% and at 2% of strain) are shown in Fig. 2. The NC metal (N1) shows the strain softening behavior during 30 tension cycles and strain hardening behavior during 30 compression cycles for all regimes. At the end of HCV deformation after 90 cycles the NC copper shows identical stresses as at tension as well as at compression cycle. The NC metal after heat treatment up to 200 °C with low heating rate at 1 °C·min^{-1} (N2) shows strain hardening at compression during first regime and during next 60 cycle's has stabile values of stress amplitude for compression as well as for tension. After 60 cycles this metal (N2) shows only very small softening behavior during tension cycle. At low temperatures heat-treated NC materials (N2) show high stability of mechanical properties and also fully elastic behavior during HCV deformation. The NC material which was double heat treated up to temperature 400 °C with heating rate of 2 °C min^{-1} (N3) shows the strain hardening both on tension and compression during 60 cycles. For comparisons the conventional cold-drawn CG copper (N4) was HCV deformed. It showed the large softening behavior during first 30 cycles (1% strain). During the sequence (from 1% to 2% of strain) this materials showed a stabile stress behavior during 30 cycles of tension as well during 30 cycles of compression. After 60 cycles the CG copper (N4) showed softening behavior only and the ultimate tensile stress decreased from 300 to 210 MPa during HCV deformation. The annealed CG metal (N5) showed during HCV defor-

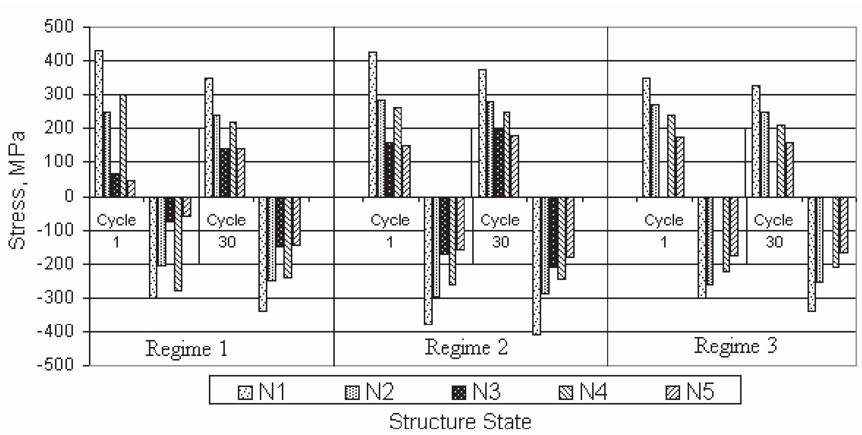

Fig. 2. The maximal values of axial tension and compression stresses at cycle 1 and at cycle 30 for regimes 1, 2, and 3 of specimens N1, N2, N3, N4, and N5.

mation only hardening behavior during firsts 60 cycles at tensions and as well as during 60 compression cycles. If the cycle number increased over 60 this material (5) showed the strain softening behavior only. As it has been demonstrated on Fig. 2, the coarse-grained (CG) cold-drawn copper specimen (N4) showed during first regime approximately identical level of strength properties with NC heat treated metal (N2). During 2- and 3-regime the cold-drawn copper specimen (N4) exhibit strain softening only while the NC copper specimen N2 preserves its properties. Double heat-treated specimen (N3) and CG annealed metal (N5) showed approximately identical strain hardening behavior during first 30 cycles. By cycles number increase the UFG copper (N3) stress amplitudes are higher.

These HCV test results show that the NC metals have generally higher values of viability then the CG metals have. Unfortunately, the NC copper thermal stability is lower. HCV test results at first regime sows that CG metal after annealing at temperature of 650 °C for 1.5 h has identical influence on maximal values of stress amplitude as the double heat treatment up to temperature of 400 °C with heating rate of 1 and 2 °C·min^{-1} has.

In addition to HCV deformation the specimens were subjected to quasistatic tensile test to study the influence of HCV deformation on ultimate tensile strength, the influence of HCV to the microstructure, the micro voids formation and fracture mode at tension testing. The tensile strain-stress curves plotted in Fig. 3 were used for viability analysis. It can be seen in Fig. 3 (curves N1 and N6) that the ultimate tensile stress decreases and the total elongation increased due to the HCV deformation. The total elongation of the annealed material subjected to HCV deformation decreased down to 10%. The ultimate tensile strength was reached under 40% of strain. The true stress at fracture did not change. The NC metal (N2) after low temperature heat treatment for structure homogenization showed very high true stress value, up to 1650 MPa [11]. In region of necking the grains coagulation to subgrains and microvoids forming took place. HCV deformation influenced to decrease (with comparison of initial NC metal) of Young's module up to 3 times.

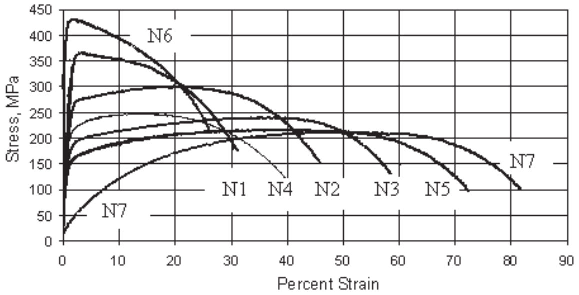

Fig. 3. Stress-strain curves after HCV deformation of specimens N1, N2, N3, N4, and N5 are shown. For comparison test the stress-strain curves of specimens N6 (NC copper after SPD of ECAP of 11 passes) and N7 (CG annealed at 650 °C for 1.5 h) without HCV deformation are shown.

4. CONCLUSIONS

The test results show that significant changes in physical and mechanical properties of a metal during the short-time HCV deformation on test; thus the test method is quite effective. The optimal HCV deformation testing parameters are: 1 – strain amplitude of 1 – 2% as optimal; 2 – strain controlled regime for 20-30 cycles during 600 s (10 min); 3 – deformation speed as short as possible – one cycle of deformation for 20 seconds (deformation speed is 0.0125 mm s^{-1} for amplitude of 1 % and deformation speed is 0.025 mm/s for amplitude of 2 %); 4 – to avoid the buckling of the specimen during alternative loading a tensile-compressive test specimen with reduced length should be used, i.e. the ratio between length and diameter of a sample is below three.

Being compared with LCF and HCF testing techniques, HCV deformation testing method is very short in time; 20-30 minutes are needed to study the viscoplastic and viability properties of the cyclically heavily loaded metal.

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