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NEW ADVANCED TECHNOLOGIES FOR NANOCRYSTALLINE METALS MANUFACTURING

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Abstract: *This critical review describes new advanced technologies for metals and alloys structure and properties directional change by sever plastic deformation (SPD) techniques*
In this study, among various SPD techniques, the equal channel angular pressing (ECAP) for material structure evolution up to nanocrystalline was chosen. The results of present investigation show that this method is very effective for producing of ultra-fine grained and nanocrystalline structures in the large billets for work mechanically or heat-treat. The nanocrystalline structure materials with exclusive high mechanical properties and hardness in future in different advanced mechanisms or installations can be used by low temperatures. For compare the electric upset forging technique (EUF (titanium and nickel based alloys treatment and machining) was used.

Key words: *Severe plastic deformation, nanostructure materials.*

1. INTRODUCTION

For manufacturing of nanostructure materials the severe plastic deformation (SPD) method (Segal, 1977) was invented. Later the apparatus and method for deformation of metals, ceramics, and other materials (Segal, 1995) was invented. During the last decade the methods and apparatus of SPD techniques were studied and invented by: (Valiev, Zhu & Lowe, 2000) the equal channel angular pressing (ECAP), (Jiang, 2000) high pressure torsion (HPT), (Tsuji, 1999) accumulative roll bonding (ARB), (Lowe & Valiev, Ghosh & Huangl, 2000) multipass coin-forging (MCF), (Zhu, 2001) repetitive corrugation and straightening (RCS), (Saito, 2000) conshearing process (CP), and (Lee, 2002) continuous confined strip shearing (C2S2) processes. By this processes use the metals structure and properties can be change step-by-step during processing. Depending on materials mechanical characteristics and manufacturing techniques the cold, warm or hot condition of processing can be used.
The effectiveness of ECAP for large billets with ultra-fine grained (UFG) structure for future processing is more conventional. According this process the cylindrical or square bar pressed by low speed on hydraulic press through an ECAP die. This die has two equal cross section channels that typically intersect at 90°. By this technique the passes number, temperature and the route mode are important parameters. These parameters control the degree of grain refinement at material. The homogenous nanostructure with high-angle grain boundaries can be formed after eight-nine passes. The deformation hardening of metal is possible after one-two passes of ECAP, but by this the microstructure of metal has very directional character. The direction is according to metal flow by SPD. Grains reduction took place during SPD at shear strain in the ECAP deformed region (Kommel et al., 2004). By SPD techniques the materials grains reduction took place from shear stress at single action of deformation only. Reduction in size can be accomplished by crushing. For material structure transformation to nanocrystalline (by ECAP) is necessary up to ten ECAP passes, depending on processing routes mode and temperature.
High pressure torsion (HPT) is more effective for structure transformation (Zhilyaev et al., 2003 & Valiev et al., 2002), but this process can by used only for very small specimens only. Dimensions of specimen are: 20 mm in diameter and thickness up to 2 mm.
For metal plate's structure forming the accumulative roll bonding (ARB), multipass coin-forging (MCF) and respective corrugation and straightening (RCS) (Huang, 2001, Shin, 2002, Huh, 2002) techniques can by used. Unfortunately, the technology is too complicated to manufacturing.
Metal sheets' forming is possible by conshearing process (CP) and continuous confined strip shearing (C2S2). These processes are effective for light-gauge fine-grained metal manufacturing.
Was shown (Kommel et.al., 1998) that for grains refining of titanium alloys is very effective the two action process, include rapid electrical contact heating (ECH) with subsequent SPD. This process is defined as electrical upset forging (EUF) technique. Effectiveness of EUF is embedded in rapid electrical conduction heating (ECH) and angular severe plastic deformation of heated up to deformation starting temperature of metal during one route. Cumulative effect from these influences on structure transformation ways is different. The pathway of the transformation may be visualized as the sum of two reactions. At first, the grains were finely divided from destructive stress at ECH. Destructive stress in grain increase according to of electrical current density increase. The deformation rate has influence on structure transformation, but main parameter is the deformation starting temperature. The structure resists the energy that acts from the outside. So, by EUF technique the grain reduction took place by the cumulative effects: density of electrical current/heating rate at first and the angular severe plastic deformation of metal after. Furthermore, during ECH the heating speed of metal increase up to 250°C/sec and as result of this the polymorphic phase transformation temperature of titanium alloys increase on 150-200 °C, respectively. This temperature of transformation increase depends on retarding of the diffusion processes, who initiates the increasing of critical temperature of hardening. When during ECH the heating speed was increased only over 100°C/sec the critical temperature of titanium increase up to 50-60°C. For comparison, in previous investigations (Kopylov, 2000) of SPD nanocrystalline materials was shown, that their mechanical properties increase and unfortunately, heat proof properties decrease by grain size decrease for all metal materials.
Among various techniques in this critical review, the ECAP for large cylindrical specimens manufacturing from other SPD techniques was chosen. For compassion the two-action process, so-called EUF was in the view-point of fitness to work; their mechanical properties stability during life-time at high temperatures were compared.
In present time the high mechanical properties preserve of SPD nanostructure materials is very complicated. By this the structure transformation during HCV deformation (Kommel, 2004) and during heat treatment influence is complex too.

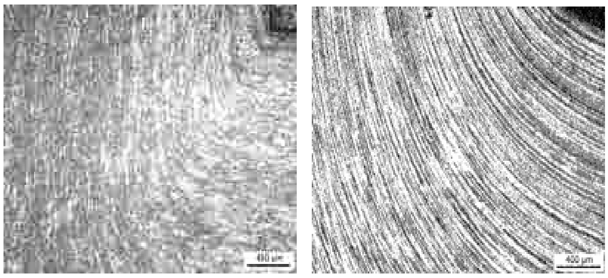
In this study the ultra-fine grained and nanostructure metal materials processing techniques are shown and materials properties received and compared. Exactly the effectiveness of ECAP and EUF techniques for metallic materials structure transformation and properties are studied and compared. By this, the optimizing of EUF and SPD processes parameters and minimizing of SPD processes cost, and effective using of nanostructure advanced materials are actual at present time.

2. EXPERIMENTAL

The ECAP process features were shown in my previous works (Kommel et.al., 2004). After ten passes of ECAP by route B_c the nanostructure in pure copper was formed. The material has high mechanical properties, such as tension stress, hardness, low softening rate during ratcheting and HCV deformation.
The EUF process features, structure transformation and formed properties influence on life extension by high cycle fatigue (HCF) were shown in my previous works (Teterin et al., 1998, Kodess, et al., 2000 and Kommel, 2001). Were investigated the HCF of turbo-jet compressor blades, manufactured from heat-proof titanium (Ti7Al4Zr4MoSnW) and ferriferous nickel (Ni28Fe16Cr14Mo3W2TiAlBP) superalloys. Was shown (Kommel et al., 1998), the blades life extension was increased up to 3-5 times with comparison of blades life extension of base manufacturing technology.
To find out changes in materials properties the indentation method on universal hardness tester Zwick Z2.5/TS1S was used. The microstructure of materials was determined by means of optical (OM) Nickon CX and scanning electron microscopes (SEM) Gemini LEO Supra-35. The low cycle HCV deformation of copper standard specimens was conducted by means of INSTRON 8516 installation.
The blades were tested on HCF and life extension on electrodynamics' vibration stand by room temperature by frequency at 1000-1180 Hz self-fluctuations and corresponding loading of blades. The one step duration of HCF test was $2x10^7$ cycles by constant load. After each step of testing without crack forming or total fracture of blade the load was increased on 40 MPa and the testing was carry a test to crack forming or total failure of a blade. For strain measurement on the testing parts of blade material (on distance at 5 mm and 15 mm from root) the extensometers were used.

3. MECHANISM OF STRUCTURE TRANSFORMATION STUDY

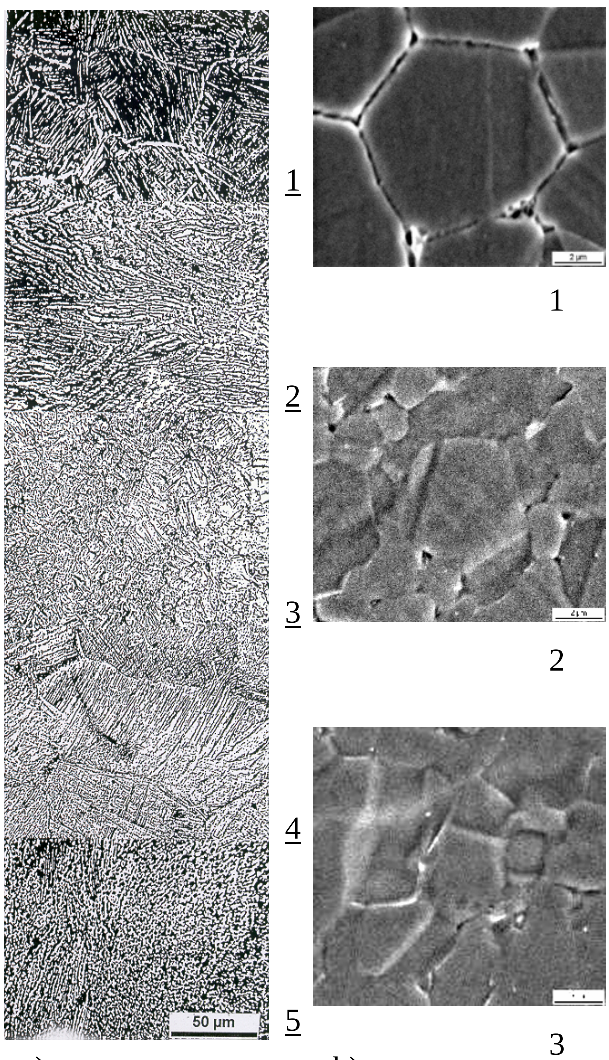
At first, the structure transformation region during ECAP and EUF processing's were investigated. Results show, that by ECAP by angle 90° between channels in the die the thickness of this region is up to 4-5 mm (Fig. 1, a) and the structure transformation can be accomplished by crushing at shear stress of grains, their thinning took place.
By EUF the deformation region is identical in view (Fig. 1, b). In this case the angle contained by directions of metal plastic flow in region. Metal flow turn through an angle of about 45 degrees. This angle depends from process parameters. The properties of the material change in going from initial diameter to entrained diameter or radius of transition. The details of structural features (between ECAP and EUF) contained in the high speed of heating by ECH method. For EUF this heating speed may be increased up to 250°C/sec. This heating speed depends on material physical properties and density of electric current in the specimen. The temperature of deformation starting and maximal temperature is difference. The starting temperature of deformation depends on deformation stress and mechanical properties at this temperature of metal. The maximal temperature of the difference parts of specimen to a considerable extent from deformation stress and degree of deformation.



a) b)

Fig. 1. Microstructure transformation region by SPD is shown. ECAP deformed region by angle 90°, (a), EUF deformed region by angle 45°, (b).

Structure investigations of titanium alloy show that at high heating speed the large grains (Fig. 2, a-1) were at first thermal decompositioned (Fig. 2, a-3) and at second deformed up to ultra-fain grained (Fig. 2, a-5) microstructure. By low heating speed and temperature the large grains were deformed only (Fig. 2, a-2). By low deformation stress the temperature can be increased over critical temperature and the cast microstructure was formed (Fig. 2, a-4). The EUF processed ferriferous nickel based superalloys microstructure investigation shows (Fig. 2, b) the grains reduction in size and grain boundaries change up to confluence of smaller grains.



a) b)

Fig. 2. Microstructure transformation by EUF is shown. Ti-alloy (a-column), and Ni-based superalloys (b-column).

At first, the lacks of structure can be eliminated. The cracks can be welded; grain in size reduced, chemical content of grains and grain boundaries changed, and activates an electron to a higher level etc. At second, depending on deformation temperature the grain size can be increased or decreased (Fig. 2, a). At all, the grain size increasing took place when heating rate is low and heating temperature is higher. The grain size decreasing took place when the heating rate is above 100°C/sec and higher. The fine-grained structure formed at high pressure stress or lower deformation starting temperature. All these parameters depend on materials physical or thermo-dynamical properties such as conductivity, ultimate-compressive strength at high (die forging) temperature, absorption of heat, thermal conductivity, heat capacitance of material, runaway of heat, heat transfer, contact conditions of specimen to electrode, etc.

4. INFLUENCE OF (MICRO-, NANO-) STRUCTURE STATE ON PROPERTIES OF METALS

All theories of viscoplasticity (Scheidler et al., 2003, Chun et al., 2002, Kenk, 2001) of materials were numerically simulated, but in this studies were don't take in account the micro-, nano-structure state of materials. Only present days in investigation (Kenk et al., 2003, Kommel, 2004) of viscoplastic behavior of pure copper of various structures the differences between cold-drawn, annealed and nanocrystalline structure test results were shown. The cold-drawn copper show (Fig. 3) only softens behavior. The annealed copper shows deformation hardening during first sixty cycles and then the softening by cycle's number increase took place. By this the nanostructure material after test starting show during ten cycles the hardening at compression and softening at tension. The cycle's number increase presented of nanomaterials stability by low cyclic deformation. They show approximately elastic behavior as rubber (Kommel et al., 2002).

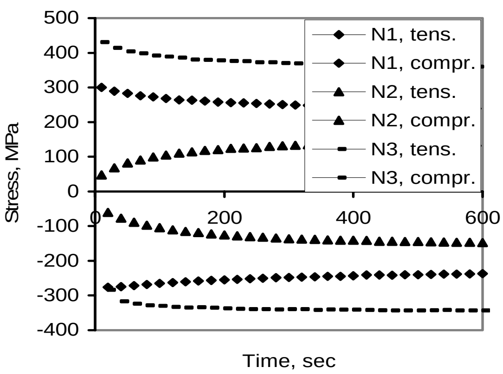


Fig. 3. Effect of HCV (by 1% of axial deformation) on deformation hardening-softening of copper by tension and compression: N1-cold-drawn, N2-recrystallized, N3-nanostructure state.

The high cyclic fatigue (HCF) and life extension are very important parameters of materials for turbo-jet compressor blades manufacturing. The SPD nanostructure materials high strength properties decrease at temperature increase. This is a lack of all nanostructure materials fabricated by all SPD techniques (Kopylov, 2000). In investigation (Kommel et al., 2004) was shown the relaxation increase in the ECAP deformed region by deformation temperature increase. These materials need for properties stabilize the heat- or deformation treatment (Kommel et al., 2004). At present time these nanomaterials don't use as materials for high temperature application.
This lack of materials can be eliminated in case when the EUF process was used. As was shown (Kommel et al., 1998), by EUF the two actions of process influenced not only on structure transformation of material but then on mechanical properties. By indentation testing method were established (Kommel et al., 2001), that the indentation work was increased. Exactly the elastic component was increased up to two times with comparison of starting state of Ni-based superalloy. After EUF the elasticity and indentation module were on level of monocrystalline state superalloy. By this during ECAP the hardness and indentation module of materials were increased and on opposite to EUF. The EUF processed Ni-based superalloy established, that the creep is lower at 10% and relaxation is lower at 15% then monocrystalline materials show (Fig. 4.). During ECAP processing the creep and relaxation were increased on opposite.

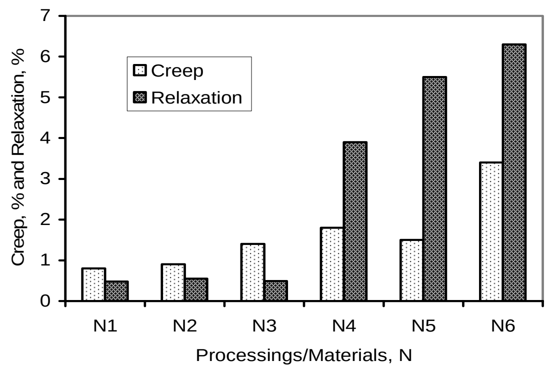


Fig. 4. Creep and Relaxation of materials vs. manufacturing technologies: N1-EUF processed Ni-based superalloy, N2-Extruded and die-forged Ni-based superalloy, N3-Cast monocrystalline Ni-based superalloy, N4-Cold-drawn copper, N5-Recrystallized copper, and N6- ECAP processed nanocrystalline copper.

The comparison tests show, that during EUF processing and ECAP processing the materials mechanical properties change took place, their change tendency is opposite. The changes of microstructure vs. mechanical properties have influence on HCF properties and HCV deformation (Fig. 3) of all materials.
The comparison test results show, that EUF processing have influence on life extension increase and high cycle fatigue (HCF) of Ti-based and Ni-based superalloys: the materials for aerospace application. The ready-made compressor blades from these materials for turbo-jet were tested on electrodynamic's vibration stand at room temperature by frequency at 1000-1180 Hz self-fluctuations and with loading of blades. Test results show that the stress levels by σ_{-1} increase from σ_{-1} = 360 MPa up to σ_{-1} = 600 MPa in maximal. Were established (Kommel et al., 1998), that using EUF for profiling performs from Ni-based superalloy EP-718id for precise isothermal stamping blades of high-pressure double-contour compressors of turbo-jet engines gives rise of blade fatigue strength by the factor of 1.3 till 1.7 as compared with that obtained after forging performs in drop forging crank press without or with high-temperature thermomechanical treatment as well as after hot extrusion followed by hot roll forging. As a result of blade fatigue strength rise, engine resource (life time) increases significantly. The life time was increased up to five times in reality.

5. DISCUSSION

The microstructure observation showed that the copper microstructure and Ni-based superalloy microstructure as-well-as Ti-based alloy microstructure transformation took place in the SPD region. By this to this region by ECAP material comes at room temperature and by EUF material comes at deformation starting temperature. By ECAP the material large grains thinning took place at shear stresses only. By EUF the material comes with changed at electrical current influence and rapid heating state of microstructure. The grains reduction in size to some extent can be accomplished by crushing in these regions from shear stress (Fig. 1, a, and b). Unfortunately, the coefficient of deformation for EUF process is lower, up to 90% in maximal value for one pass. By ECAP the coefficient over one and the passes number don't limit. By this process depending on passes number and temperature the grain size can be transformed to nanomeasures. Unfortunately, the nanostructure materials don't have stabile state by temperature increase. For airspace this process can't use. For properties stabilization of nanomaterials the heat treatment with deformation can be use (Kommel, 2004).

The SPD nanostructure materials high tension stress is favorable. The EUF processed materials have fine-grained structure (Fig. 2, a, and b) without cracks or any defects. After EUF the Ni-based superalloy erosion between grains wear is lover with comparison of state material after state treatment.

6. CONCLUSION

In this critical reviews which advance the understanding of the standard and functional properties of materials vs. fabrication technologies: ECAP and EUF was shown. Depending on needed materials with special exploitation properties the technology process and process parameters are choose in preview. ECAP processed nanomaterials have high tension stress and high hardness but lowest thermal stability properties highest and relaxation. For thermal stability of properties the low temperature heat treatment is needed.

EUF processed materials hardness and indentation module decrease, and elasticity properties increase for all metals. The creep and relaxation are lowest for EUF with comparison of other manufacturing techniques. They have excellent high life extension at HCF and thermal stability properties. EUF processed metals can be use at high temperature application. The EUF technique delivers more effective by comparison with SPD techniques for titanium alloys hot working.

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