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EVALUATION OF DRECE FORMING PROCESS OF CU-BASED ALLOYS AND PROPOSAL OF TESTING METHOD FOR EVALUATION OF UFG MATERIALS MICROSTRUCTURAL STABILITY

VYHODNOCENÍ ZPRACOVÁNÍ PÁSŮ SLITIN NA BÁZI CU PROCESEM DRECE A NÁVRH ZKUŠEBNÍ METODIKY PRO HODNOCENÍ MIKROSTRUKTURNÍ STABILITY UFG MATERIÁLŮ

Abstract

The main goal of the paper is a review of current achieved results given by processing of Cu-based materials by DRECE technology, whose prototype equipment has been put in service in the end of 2008 at VSB-Technical University of Ostrava, Faculty of Mechanical Engineering and Dept. of Mechanical Technology. Two types of Cu-based materials – technical pure Cu (99,5 % purity) and brass in α modification – have been used like an experimental materials. The paper describes results of determination of an influence of pass number on output mechanical and micro structural properties of processed Cu-based materials. The paper shows some demonstration of a micro structural evaluation with using of light microscopy and TEM. The paper also shows proposals for testing procedures for determination of UFG materials micro structural stability after ECAP process by AlMn1Cu and AlFe1,5Mn alloys. First method is based on determination of critical temperature given from observed changes of mechanical properties. Other possible method, more valuable form a view of technological use, is based on physical description of grain-coarsening processes.

Abstrakt

Příspěvek uvádí současně dosažené výsledky se zpracováním materiálů na bázi Cu tvářecím procesem DRECE, jehož prototyp zařízení byl na konci roku 2008 zprovozněn na Katedře mechanické technologie, FS, VŠB-TUO. Jsou zde uvedeny výsledky stanovení vlivu počtu průchodů na výstupní mechanické vlastnosti a metalografické studium zpracovávaných materiálů. Příspěvek dále uvádí navržené postupy hodnocení mikrostrukturní stability UFG materiálů po procesu ECAP metodou stanovení kritické teploty z průběhu sledovaných změn mechanických vlastností - a možný návrh dalšího, z hlediska technologického uplatnění výhodnějšího, zkušebního postupu hodnocení, vycházejícího z fyzikálního popisu procesu hrubnutí zrna u slitin AlMn1Cu a AlFe1,5Mn.

1 INTRODUCTION – USED DRECE TECHNOLOGY

Used DRECE technology belongs to group of a progressive type of forming processes making use of severe plastic deformation (SPD). The SPD methods are enable to process suitable materials to achieve ultrafine grain structure (UFG) with mean grain size c. 1μm [1]. After this processing the materials exhibit – in comparison with conventional structure materials – significant higher mechanical values – especially yield strength, in limited also ultimate tensile strength.

Forming process DRECE is an extrusion technology with limited cross-sectional reduction to achieve high degree of deformation of suitable selected material. A prototype of this equipment has

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been put into use from the end of year 2008 at workshop of VSB-Technical University of Ostrava, Faculty of Mechanical Engineering and Dept. of Mechanical Technology.

Prototype of the equipment is shown at fig. 1.

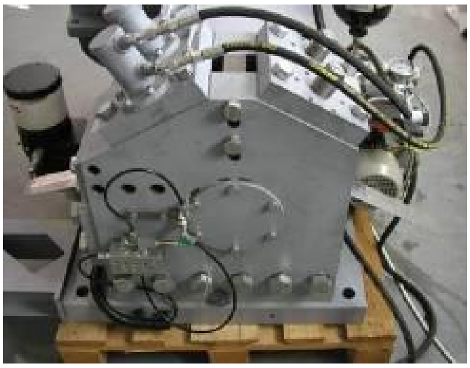

Fig. 1: DRECE prototype equipment - generally view

During testing period it was necessary to solve some problems about progress of the forming process – to solve problems about finding a suitable surface roughness of support cylinders, then to reduce warp and jamming of sheet during the process. Also a new geometry of forming tool has been designed – see fig. 2a, b [2].

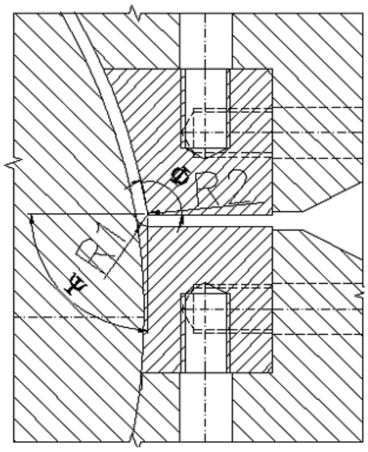

Fig. 2a: Original geometry of DRECE forming tool

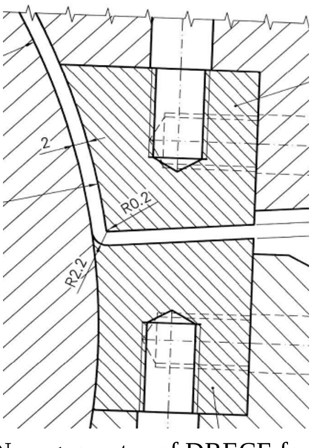

Fig. 2b: New geometry of DRECE forming tool

Series of mathematical simulation of deformation were performed before making the new forming tool geometry. The simulation of sheet made of Al 99,5 with using Simufact Forming program was performed for design study of the forming tool after fulfilment of all set conditions. The conditions are following: Φ = 100°, outer degree radius Ψ = 0°, radius $R1$ = 10 mm and $R2$ = 10 mm. Part to be processed (sheet) is divided into 7302 elements of dimension 5 mm. HEXAHEDRAL type of the element and SHEETMESH netting have been selected. Demonstration of the deformation simulation after first and second pass at mentioned conditions are showing a fig. 3a, b.

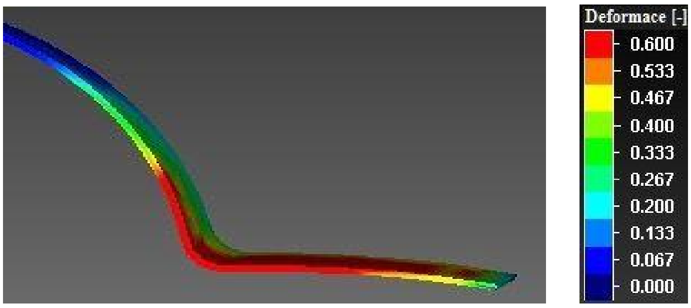

Fig. 3a: Stress intensity during 1st pass

Deformation intensity after first pass achieves a maximum of ε = 0,6. Stress intensity after first pass achieves a maximum of σ_i = 150 MPa [2].

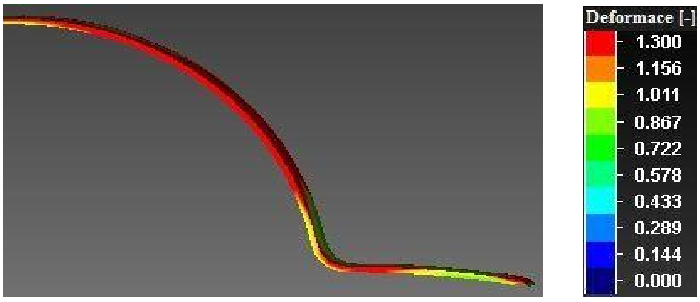

Fig. 3b: Stress intensity during 2nd pass

Deformation intensity after first pass achieves a maximum of ε = 1,3. Stress intensity after first pass achieves a maximum of σ_i = 185 MPa [2].

All the works related to testing period of the prototype had been performed during year 2009 and now the equipment is fully used for many experiments about optimalization the forming process to ensure its high effectively and for obtaining the highest quality of material output.

2 USED EXPERIMENTAL MATERIALS

Sheets from commercial pure aluminium (99,5 Al) of dimensions (55x2x1000) mm – width x thickness x length – were used for very beginning experimental works. This material was used for practical solving of first technological problems with jamming and high warp of processed sheets. But it is not taken account of this material any more for next experiments due to its poor increasing of mechanical values [4]. Cause of this behaviour is most likely because of low recrystalisation temperature of the material that could be locally exceeded during the forming process.

Cu-based materials have been selected for next experiments after performed literature search and getting up a binary phase diagrams (in the case of alloys).

It is a:

- commercial pure copper (99,5 Cu),
- brass in α modification.

As well as in previous case of commercial pure aluminium - a sheet of dimensions (55x2x1000) mm – were used like an input material. The sheets have been bought in commercial trade network in as cold rolled state, without previous heat treatment, in chemical purity corresponding to relevant standard.

3 DRECE FORMING OF THE EXPERIMENTAL MATERIALS

Both the experimental materials have been formed by DRECE process at ambient temperature, without previous heat treatment and operative heating between individual passes. The sheets were 180° rotated around its longitudinal axe between individual passes.

Performed pass numbers:

- Cu 99,5: 2x - 4x - 6x - 8x - 10x
- α brass: 2x - 4x – (6x presently performed)

DRECE processed sheets were given to metallographic evaluation and for determination of basic mechanical properties.

Remaining sheet were further heat treated at 450 °C/15 min/air (without protective atmosphere) for evaluation of an influence of used operative heat treatment on an output properties of used materials. These results will be available in fall 2010.

4 METALLOGRAPHIC EVALUATION

Mentioned photos have been made at workshop of metallographic laboratory of VŠB -TUO. Parallel works are also performed at workshop of Laboratories and testing shops of VÚHŽ a. s. Structure etching of both experimental materials were performed without using of an electrolytic etching method.

Cu 99,5

Fig. 4 shows macro photo of metallographic samples of pure copper in an initial state and after 8^{th} pass.

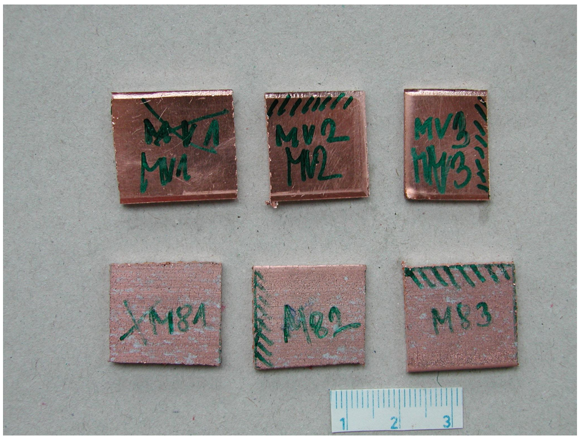

Fig. 4: Macro photo of Cu 99,5 in an initial state and after 8^{th} pass

Fig. 5 a, b show microstructure of Cu 99,5, cross-section in an initial state and after 8th pass.

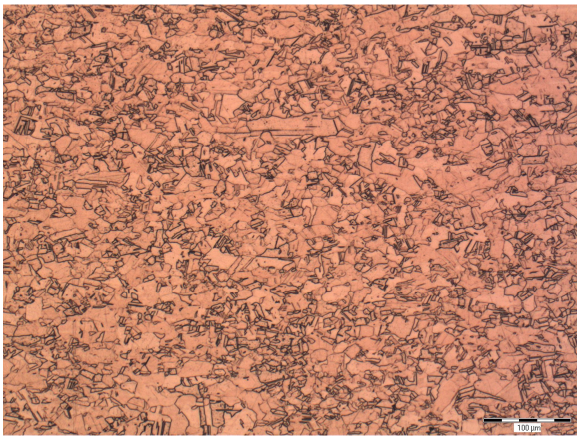

Fig. 5a: Microstructure of Cu 99,5 in an initial state, transverse direction, mag. 100x, scale 100 µm

Fig. 5b: Microstructure of Cu 99,5 after 8th pass, transverse direction, mag. 100x, scale 100 µm

Fig. 6 shows TEM sample after 6th pass.

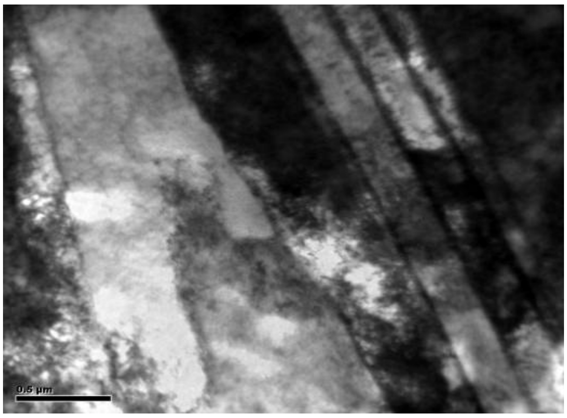

Fig. 6: TEM of Cu 99,5 after 6th pass, , scale 0,5 µmα brass

Fig. 7a, b show alpha brass microstructure in an initial state and after 4th pass.

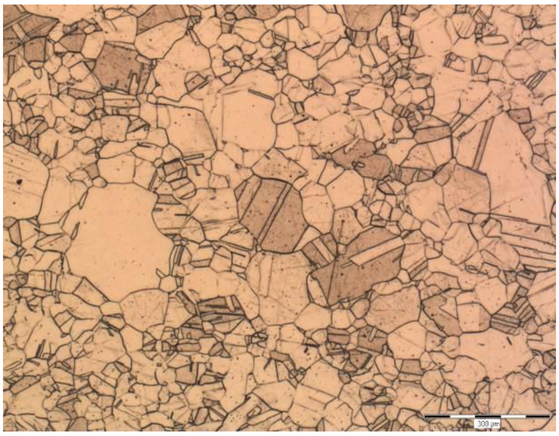

Fig. 7a: Microstructure of alpha brass in an initial state, transverse direction, mag. 100x, scale 100 μm

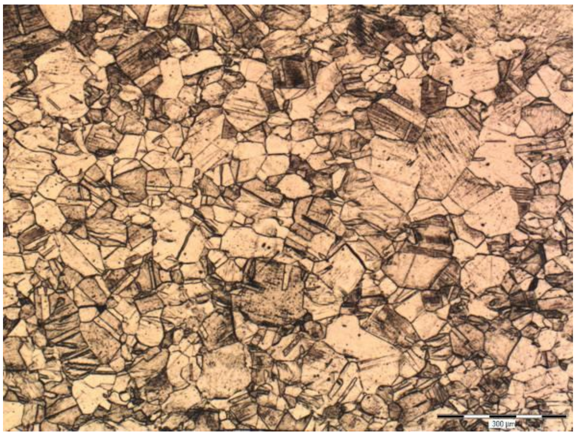

Fig. 7b: Microstructure of alpha brass after 4th pass, transverse direction, mag. 100x, scale 100 μm

5 EVALUATION OF BASIC MECHANICAL PROPERTIES

Evaluation of basic mechanical properties was performed by mechanical testing laboratory of VÚHŽ a. s. It was performed Vickers hardness measurement HV10 (HV5) according to the ČSN EN ISO 6507-1 and tensile test at ambient temperature according to the ČSN EN ISO 6892-1 (in accordance with requirements of Annex B).

Standardized test-pieces (type II) of dimensions – a_o = 2,0 mm, b_o = 12,5 mm, l_o = 80 mm – were used for all the tensile tests. Used stress rate 20 MPa.s^{-1} within elastic deformation zone, used deformation rate 0,0024 s^{-1} within strain hardening zone – designation ISO A3B10 [3].

Fig. 8 and 9 show graphic results of determination of an influence of pass number on basic mechanical properties of Cu 99,5 and α brass.

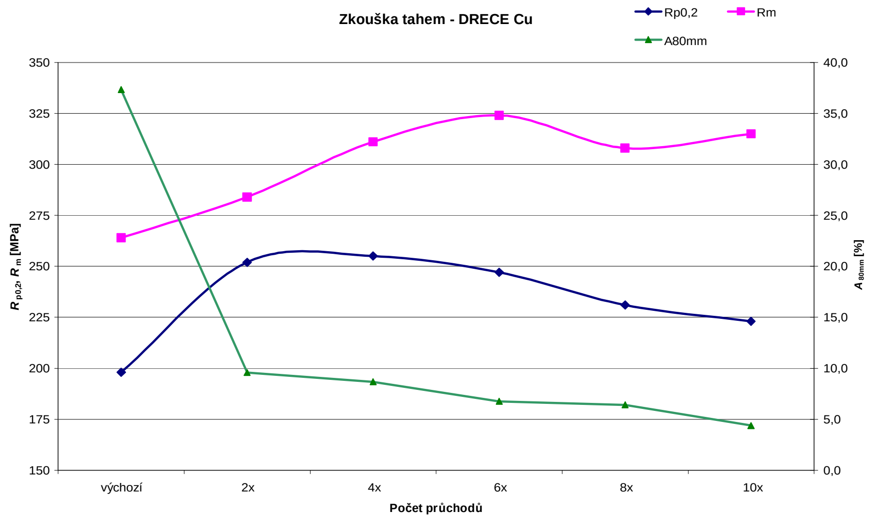

Fig. 8: Influence of pass number on mechanical properties of Cu 99,5 (tensile test at ambient temperature)

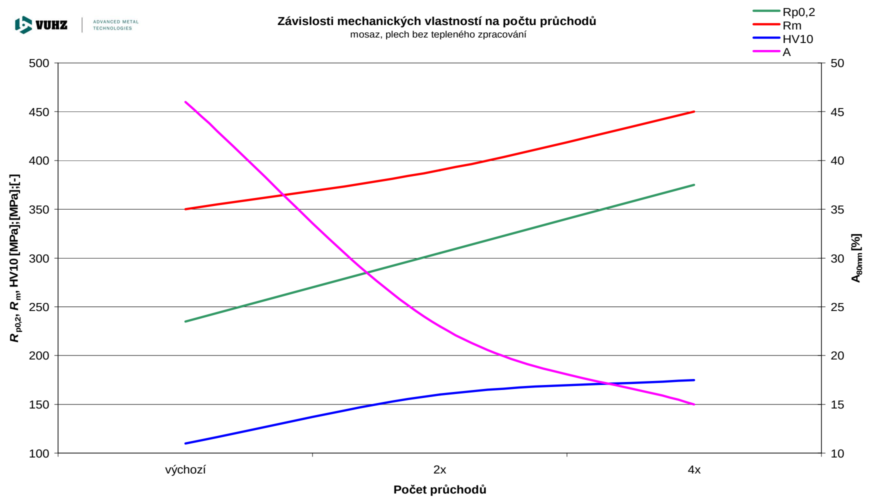

Fig. 9: Influence of pass number on mechanical properties of alpha brass (tensile test at ambient temperature, Vickers hardness)

The results present exhibit positive influence of performed DRECE process on an output basic mechanical properties. It is observed that there is a uniform increasing of yield as well as tensile strength at given initial micro structural state. Also observed ratio $R_{p0,2}/R_m$ between individual passes is about constant - i. e. favourable effect from a view of achieving suitable ability to strain hardening behaviour within a zone of nonreversible (plastic) deformation.

There is observed maximum at yield strength of processed Cu 99,5 after c. 4 passes. Further increasing of passes does not bring a significant increasing of yield and tensile strength. In comparison of both used materials, alpha brass seems a more suitable for DRECE processing due to uniform increasing of mechanical values and its saturation could be expected after more than stated 4 passes with 180° rotation between individual passes.

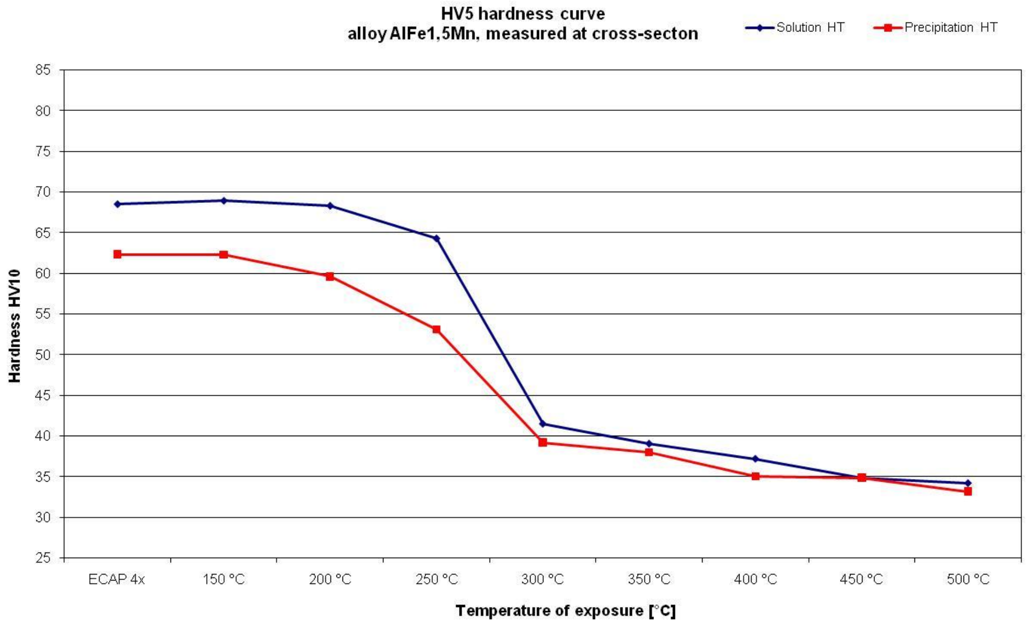

Fig. 10: Determination of a critical temperature of AlFe1,5Mn, note: S – solution annealing, T - precipitation annealing

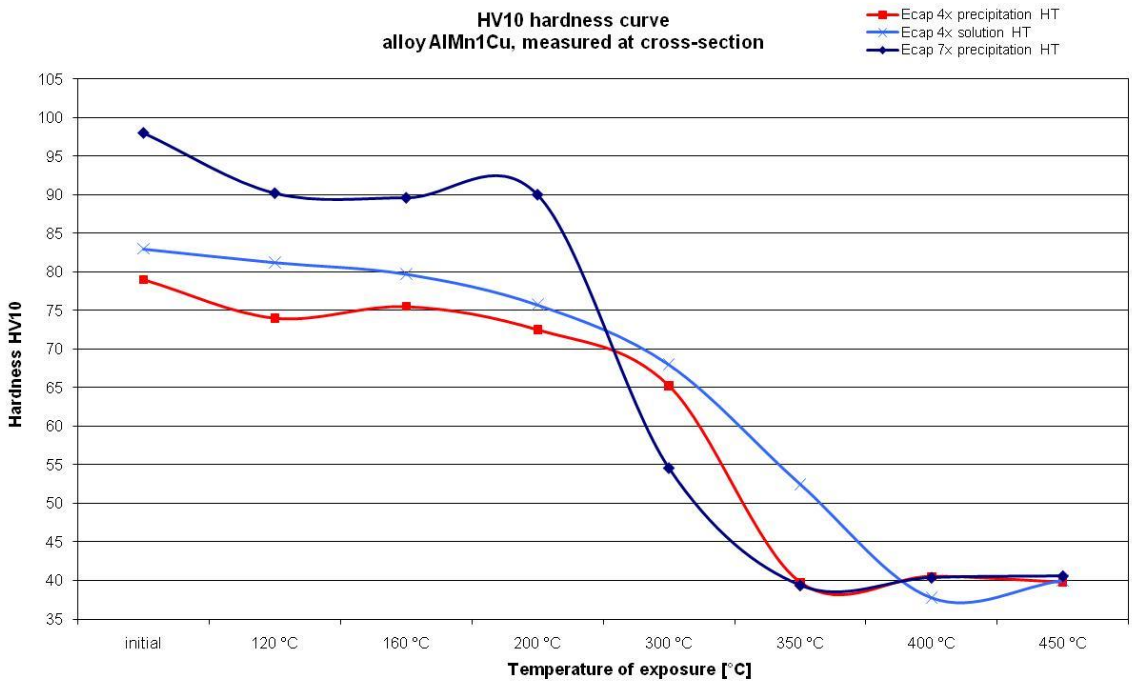

Fig. 11: Determination of a critical temperature of AlMn1Cu, note: solution annealing, precipitation annealing, 4x, 7x - number of passes

It could be observed from given results that the critical temperature at the same testing conditions is about 250 °C in a case of AlFe1,5Mn alloy and it is about 300 °C in a case of AlMn1Cu alloy [5]. Even these simplified short-time tests are able to inform about expected micro structural stability that is necessary for example for finding if the UFG material could be processed by such procedures like soldering in continuous furnace, which is usually used for fabrication of automotive air-condition parts [5].

Above mentioned testing procedure is very simplified approach that assumes only constant dwell time, i. e. results are usable only for observed conditions. So it has been proposed one more potential testing approach that could include required time view as well as determination of material stability from a view of degradation process activation energy.

The main thesis is based on next approach:

- Micro structural changes process are primarily controlled by grain coarsening process, i. e. by secondary recrystalisation and there is no significant influence of previous processes of grain recovering
- it is assumed that the effect controlled only by diffusion
- parameter to be observed is mean grain size (it is necessary to establish conventional testing procedure to determine it due to high grain size inhomogenity)

Practicability of the testing approach is now evaluated on ECAP processed AlFe1,5Mn alloy. It is necessary to perform complete TEM analysis that confirms/disconfirms that the leading process is secondary recrystalisation. As well as to find a way how to determine representative mean grain size. So if the approach will be find real, micro structural stability evaluation will performed on the basis of next procedure:

It is possible to describe a grain coarsening process by Arrhenius type equation that includes influence of temperature, as well as influence of dwell time of exposure.

$$_{\Delta}d = A * t * \exp\left(-\frac{Q}{RT}\right) \tag{1}$$

where:

$_{\Delta}d$ - change of mean grain size [μm],
A - material constant [μm/s],
t - dwell time [s],
Q - grain coarsening activation energy [kJ/mol],
R - gas constant [kJ/(mol.K)],
T - thermal exposure temperature [K].

It means that is necessary to find describing material constants for micro structural stability evaluation in general. Constant A describes material from a view of grain coarsening kinetics and constant Q describes the threshold energy need for starting the grain coarsening process.

Testing procedure in general:

- *microstructure homogenization by suitable heat treatment, determination of mean grain size*
- *SPD processing, metallographic evaluation, determination of mean grain size*
- *series of thermal exposure at constant temperatures and different dwell times*
- *metallographic evaluation, determination of mean grain size*
- *evaluation of testing results (determination of Q and A) – see bellow*

Determination of material constants is based on general description of micro structural changes kinetics of diffusion effect – it means it is calculated with temperature as well as time aspects.

Determination of Q and A constants are performed by two individual testing procedures.

- determination of grain coarsening rate

It is finding by next procedure:

Performing of an isothermal exposure at different dwell times – for each series is to be determinate grain coarsening rate in dependence on dwell time. Selection of exposure temperature could be based on testing results given by determination of critical temperature – see above.

According to:

$$v = \frac{\Delta d}{\Delta t} \tag{2}$$

i. e. scope of curve in system d = f(t)$_{T = konst.}$

where:

v - mean grain coarsening rate [μm/s],
d - mean grain size [μm],
t - time [s].

And thus it is to be determinate a corresponding mean grain coarsening rate v_1 to v_n for series of isothermal exposure at T_1 to T_n.

It is necessary to perform a series of four thermal exposure (dwell time) at least. These results unambiguously describe a reaction kinetics, so observed influence of temperature and time. These are the only particular results. Determination of desirable Q and A constants is performed on the basis of results describing kinetics of the process.

- determination of *Q* and *A* constants

Determination of the constants is to be performed by least squares method and it also uses Arrhenius description.

$$v = A * \exp\left(-\frac{Q}{RT}\right) \tag{3}$$

note: designation as above

The equation is to logarithmic transformed into linear form:

$$\ln v = \ln A - \frac{Q}{R} T^{-1} \tag{4}$$

note.: designation as above

Observed material constants are determinate by substitution of upwardly ordered experimental results into (ln v = f (T^{-1})) system by least square method (determination of slope and initial point of the line).

This mentioned testing procedure evaluates micro structural stability with high predicative value. The evaluation could be performed from a view of determination of maximum allowable temperature, maximum allowable time exposure and all of this could be based on conventional requirements for UFG materials in general.

6 CONCLUSION

From achieved testing results of evaluation of influence the DRECE process on Cu based material, it stands to reason that the process is very perspective. Increasing of mechanical values is in order of tenth percents – for example observed increasing of HV 10 of pure copper is about 45 % compared to an initial state [4]. There will be observed an influence of operative heat treatment for increasing an effectivity of the forming process.

Mentioned proposals for evaluation of micro structural stability suppose relatively easy to perform testing procedures with sufficient predicative value.

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