Report on the Doctoral Thesis

MICROSTRUCTURE AND MECAHNICAL PROPERTIES OF ULTRA-FINE GRAINED MATERIALS PRODUCED BY SEVERE PLASTIC DEFORMATION

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Topicality of the research

A superplastic behaviour of metals and alloys represents a long-standing issue investigated in many leading materials laboratories all over the world. This topic attracts the interest not only because it offers many possibilities for basic studies that improve our understanding of a relationship between microstructure and mechanical properties of materials, but it also opens a rich field of applications in the recent materials technology. The long-term research in the area has shown unambiguously that grain boundary sliding is a key mechanism that, under certain circumstances, can back up ductility of the order of 10^3%. Two conditions necessary for grain boundary sliding being an efficient mechanism of superplasticity are (i) (ultra)fine grain microstructure and (ii) its stability in the range of external conditions (in terms of temperature and strain rate) where superplastic flow occurs. Meanwhile, there has also been a considerable development of methods dedicated to the microstructural refinement, particularly those based on severe plastic deformation (SPD). Thus a very basic idea of the study presented in the thesis was first to act on the chosen AA 7075 alloy and its Zr and Sc modified version using one of the SPD procedures in order to form fine, homogeneous microstructures. In the next step, the thermal stability of the refined microstructures and their prospective superplastic behaviour were investigated. The author has selected equal channel angular pressing (ECAP) performed at three different temperatures as the SPD processing route. This rather unique combination of ECAP, systematic characterization of microstructural states and a careful assessment of mechanical properties at elevated temperatures promotes this study up to an important contribution to the field of superplasticity research.

Format and elaboration

A thesis volume covers 138 pages, including 72 figures, 8 tables and 215 references to publications. The volume is reasonably distributed between an introductory part, in which the state-of-the-art is reviewed, a description of objectives and experimental procedures, a summary of results, their discussion and conclusions. Here comes one of my rare comments to the otherwise well written text. I would personally think that parts of the text on pages 57-61, related to the precipitation of $Al_3(Zr,Sc)$ phase, should have been presented in the introductory chapter and/or as a part of discussion rather then in the section on the experimental alloys and characterization methods. On the other hand, I would like to highlight quite impressive list of experimental methods and particularly a subset that has been employed in order to characterize the microstructure of the as-received alloys and their states after thermo-mechanical treatments and mechanical testing. This experimental framework truly contributes to the high scientific standard of the thesis. As it has been clearly documented in the section 5.3, the optimized ECAP processing route applied to the thermally stable Zr and Sc containing alloy resulted in a high strain rate superplasticity. The results obtained for the above Zr and Sc modified alloy thus count among the best reported in the literature to date. Therefore, it is more than obvious that the format, elaboration and the results obtained and presented in the thesis meet the targets and objectives formulated in the section 3. Last but not least, the thesis is written in an almost excellent style of English, some minor typos are summarized in the subsequent part of this report. My another general comment addresses the way in which results in the section 5 of the thesis are

presented. Even though valuable data on the ECAP refinement and the microstructural evolution during the subsequent heat treatments were obtained, they are rather scattered throughout numerous pages in the section 5. Some additional tables (and/or plots) comparing directly the behaviour of the AA 7075 and the modified alloy would considerably facilitate the understanding of the crucial achievements of the study. The same holds true for a possibility to compare directly the results obtained by means of EBSD and ACOM methods. Similarly, a comparison to already published data on the superplasticity of the Al-based alloys would also help to put the new results into a perspective of the work that has already been done.

Questions and technical comments

Scientifically, there are at least three important issues that are also relevant for the results of the thesis:

1) Many controversies in the literature exist as to whether the Hall-Petch relationship (see Eq. 1.1 in the thesis) holds true for the (ultra)fine grain materials (see e.g. recent study by W.J. Kim: Scripta Mater. 61 (2009) 625-655). These discussions arose mainly because of the classical frame for the $d^{-1/2}$ dependence of the flow stress, which assumes dislocation pile-ups against grain boundaries. These pile-ups are difficult to form in microstructures with characteristic grain sizes smaller than a certain limit. In this respect, has the author verified the Hall-Petch relationship for the ECAPed materials tested in tension at room temperature? What would be the microstructural mechanism that results in the $d^{-1/2}$ dependence for grain sizes in the range of 0.5 μm? Was the relation Eq. 2.11 fulfilled for the elevated temperature data?

2) The heat treatment of the investigated materials after ECAP provided clear evidence that the thermal stability of the Zr and Sc-modified alloy is much better than that of the parent AA 7075 alloy. Was the volume fraction and the number density of the $Al_3(Zr,Sc)$ particles sufficient to exert relevant Zener-forces to pin the grain boundary structure? What about the role of a solute drag and stacking fault energy (see e.g. the reference [207] in the thesis) in slowing down the recovery processes?

3) Data presented in Figs. 33 and 63 (parts (a) of the figures) could invoke the existence of a threshold stress dependent on temperature. This concept is quite extensively presented in the introductory chapter of the thesis (see e.g. pages 38, 43 or 45), however it is not employed any further in later sections in order to rationalize the experimental data. The question is why?

Technical comments:

1) page 14, lines 14-17	There are also other methods that are efficient in refining the microstructure, like the wire drawing or martensitic transformations in the case of ferritic steels ...
2) page 19, line 17	...*materials* ***are*** *susceptible* ...
3) page 21, line 17	...*in a many applications* ... the article does not fit the plural form.
4) page 26, line 4	...*tame*... should be ...*time*...
5) page 27, last line	...*apt*... should be rather replaced by ...***considered***...
6) page 31, line 9	The term ...*ductility*... is generally reserved for tension experiments, ...***rupture strain***... is a more suitable term for other deformation modes like torsion and compression.
7) page 31, line 29	A full stop is missing at the end of the sentence.
8) page 35, line 30	...*micro-processes*... a generally accepted term is ...**microstructural processes**...

9) page 38, line 23

A common understanding of the term *threshold stress* is that, at this stress level, a **specific mechanism** of plasticity ceases, which, however, does not mean at all that it represents ...*a minimum value necessary for the material to deform plastically*...; for instance diffusion (as a very important mechanism of high temperature plasticity) is usually considered as a threshold-stress-free process.

10) page 57, line 16

Also the term ...*coherent dispersoids*... is rather unusual. The term **dispersion strengthening** is normally used in situations where a metal or an alloy contains (small) oxide particles that are, in a vast majority of cases, incoherent with the parent lattice. This type of strengthening is particularly important in the high temperature domain since the refractory particles posses and exceptional stability and do not coarsen during high temperature exposures. Consequently, I would recommend using a term particle or precipitation structure in a relation to the $Al_3(Zr,Sc)$ phase.

11) page 66, line 1

...*misorientation of grain boundaries*... what can be misoriented are two crystallites (grains) adjacent to a grain boundary, not the grain boundary on itself.

12) page 66, ACOM

Is the ACOM technique based on the nano-probe mode of diffraction? What is the influence of the TEM foil thickness and the local foil bending on the accuracy of the measurement?

13) page 89, Fig. 49

How many frames are processed using EBSD per one material state? It can be expected that the sample size of the evaluated grains does matter, taking into account the inhomogeneity of the microstructure.

14) page 90, Figs. 51 - 53

These three microstructures are difficult to compare mutually and also to the as-ECAPed microstructures in Fig. 48 due to different magnifications used.

15) page 115, lines 10 - 16

Is there a possibility that the observed orientation gradients are associated with LAGBs rather than with individual dislocations?

Conclusion

The thesis, its format and elaboration clearly document that the author is ready for his own scientific career. The results presented in the text are of a high scientific standard and represent a valuable contribution to two research fields (i) the superplasticity and (ii) the investigations of (ultra)fine grain microstructures using modern microscopy techniques. In spite of few minor issues mentioned in this report, I highly rank the submitted work. I do recommend to the commission the thesis being accepted for a further promotion and the defence.

In Brno, on 12.10.2009