Microstructure and mechanical properties of ultrafine-grained metals processed by powder metallurgy methods

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Introduction

In the last few decades bulk fine-grained (nanocrystallite and ultrafine-grained (UFG)) materials are in the focus of materials science. This increased interest can be attributed to the high hardness of these materials compared to their coarse-grained counterparts. One processing route of UFG metals is the consolidation from nanopowders. The materials investigated in the present thesis were sintered by modern consolidation processes, such as Spark Plasma Sintering (SPS) and High Pressure Torsion (HPT). During SPS high current density pulses promote the faster consolidation, thus the time and the temperature of sintering are reduced, hereby impeding the grain growth during consolidation. The HPT is usually used for the grain refinement in bulk coarse-grained metals, however the high pressure applied during HPT also enables this method for consolidating metallic powders. Besides single phase metals, HPT has also been used to fabricate nanocomposites. The goal of my PhD work was to study the effect of the processing conditions (e.g. temperature, disperse phase addition) on the microstructure and mechanical behaviour of UFG metallic materials produced by powder metallurgy methods. An additional aim was to investigate the deformation mechanisms in UFG materials at different strain rates and temperatures. Furthermore, I examined the thermal stability of the fine-grained microstructure. Due to the small size of the samples processed by SPS and HPT methods, special techniques were required to study the mechanical properties. For this purpose, the high-temperature indentation creep equipment in Department of Materials Physics at Eötvös Loránd University was improved under my control. The renovated device enables the performance of creep measurements up to 1000 °C. The accuracy of the measured values of the displacement and the temperature are lower than 50 nm and 1 °C, respectively. My PhD work was basically an experimental research, which included the examination of grain structure and lattice defects by transmission and scanning electron microscopy, as well as X-ray line profile analysis. The mechanical behaviour was studied by quasistatic and dynamic compression tests, microhardness measurements, and creep investigations. The thermal stability of the UFG microstructures was investigated by calorimetry (DSC).

New scientific results

1) Blends of coarse Cu powder and carbon nanotubes (CNT) were consolidated by HPT at room temperature (RT) and 373 K. An additional sample was consolidated from pure Cu powder at RT, by the same way as in the case of the composite specimen. Due to the pinning effect of CNTs, the dislocation density is about three times larger, while the grain size is about half of that obtained in the sample consolidated from pure Cu powder. The Cu–CNT composite processed at RT exhibited very high hardness due to the extremely high dislocation density. Both the high stresses at glide obstacles and the small grain size may contribute to the significant twinning in the composite that was not observed in the pure Cu sample. The increase of the HPT-processing temperature resulted reduction of lattice defect density, leading to the decrease of the hardness [S1, S2].

2) The thermal stability of UFG Cu consolidated from a powder by HPT were compared to a casted coarse-grained bulk Cu processed by HPT. Despite their similar microstructures in the as-received state, their thermal stability is very different. In the consolidated Cu sample the onset temperature of recrystallization was considerably higher (~800 K) than that for recovery (~500 K), while in the initially bulk-Cu specimen the two processes occurred in the same temperature range (between 400 and 540 K). The much better thermal stability of the consolidated-Cu specimen might be attributed to impurities and oxide dispersoids which are usually unavoidable in powder metallurgy. A good correlation between the released heat measured by DSC and the change of stored energy calculated from the defect densities was obtained for both the pure and composite samples [S2, S3].

3) The addition of CNTs to Cu powder increased the thermal stability of the UFG microstructure processed by HPT, since recrystallization was not observed even at 1000 K, contrary to the sample consolidated from pure Cu powder. During high temperature annealing, the large reduction in hardness of the pure consolidated-Cu sample was caused by recrystallization, while in the Cu–CNT composites the UFG microstructure of the Cu matrix remained stable and the hardness decreased mainly due to the increased porosity [S2, S3].

4) $Mg_{95}Zn_{4.3}Y_{0.7}$ powder was consolidated by HPT at RT and 373 K. Both the initial powder and the HPT-processed specimens consisted of an icosahedral Mg_3YZn_6 phase

(I-phase) besides the α-Mg matrix. The dislocation density and the crystallite size determined in the specimen consolidated at 373 K are lower and higher, respectively, than the values obtained for the sample processed at RT, due to the easier annihilation of lattice defects at higher temperature. The relative fractions of $\langle a \rangle$, $\langle c \rangle$ and $\langle c + a \rangle$ dislocations are about 70, 10 and 20%, respectively, for both samples. The abundance of $\langle a \rangle$-type dislocations can be attributed to their smallest Burgers vector and therefore their lowest energy among the three types of dislocations. The contribution of twinning to plastic deformation during consolidation was marginal, most probably due to the small grain size [S4, S5].

5) The flow stress obtained experimentally agrees well with the value calculated from the dislocation density by the Taylor-formula, indicating that the strength in pure Cu, Cu-CNT and Mg-Mg_3YZn_6 composites is determined mainly by the interaction between dislocations. This agreement also reveals that the disperse phase strengthen the composite rather indirectly via the increase of the dislocation density. [S1, S5]

6) The mechanical behaviour of an UFG Zn processed by SPS was compared to that of a coarse-grained counterpart. In the case of coarse-grained Zn compressed up to a strain rate of ~10 s^{-1} twinning and thermally activated dislocation motion controlled the deformation. Above the strain rate of 10^3 s^{-1} dislocation drag effects controlled the plasticity. In the case of UFG-Zn the relatively large dislocation density (~10^{14} m^{-2}) and the small grain size (~220 nm) limited the dislocation velocity, yielding the lack of dislocation drag effects up to 10^4 s^{-1}. Twinning was not observed in the UFG material. In the as-consolidated state of UFG-Zn, most of $\langle a \rangle$ and $\langle c + a \rangle$ type dislocations are prismatic and pyramidal edge dislocations, respectively. During the deformation other $\langle a \rangle$-type dislocations in basal and pyramidal and $\langle c + a \rangle$-type dislocations in prismatic and other pyramidal slip systems were also activated [S6, S7].

7) High temperature creep behaviour of UFG-Zn with two different oxide contents processed by SPS was investigated by indentation technique in the temperature range of 330-360 °C. The grain size in the as-consolidated samples was about 200 nm, irrespectively of the ZnO content of the material. Recrystallization during high-temperature indentation creep was not observed, indicating an excellent thermal stability of UFG-Zn samples which can be attributed to the retarding effect of ZnO dispersoids on grain growth. The activation energies of the creep were 252 ± 25 and 211

± 19 kJ/mole for specimens with high and low oxide contents, respectively, which are much larger than the value determined for coarse-grained Zn (152–159 kJ/mole). The higher activation energy for UFG-Zn processed by powder metallurgy can be attributed partly to the effect of ZnO dispersoids. The strain rate sensitivity parameter decreased with increasing oxide content. Although the dislocation density was the same inside and outside the indented zone, the distribution of dislocations among the various slip systems was different in the indented and non-indented volumes. Outside the indented region prismatic $\langle a \rangle$ and pyramidal $\langle c + a \rangle$ dislocations were observed, while in the indented zone additional $\langle a \rangle$-type basal and pyramidal dislocations as well as other $\langle c + a \rangle$-type pyramidal dislocations were detected. This observation suggests that there is a considerable dislocation activity during high-temperature creep of UFG-Zn [S8].

Applications of the new results

The high temperature indentation creep equipment – which was improved under my control – enables the investigation of the creep behaviour for UFG materials with small dimension. In addition, I have developed a method for the determination of deformation mechanisms under the indenter. This procedure was applied successfully on UFG zinc (see thesis 7). The effect of the processing conditions on the microstructure, mechanical properties and thermal stability of metal matrix composites was revealed. My results may contribute to the development of harder and more stable composites. Some of my observations fill the gaps in the literature: for instance the evolution of microstructure in UFG-Zn during high strain rate deformation has not been studied yet.

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Publications of the Author Related to Theses

[S1] P. Jenei, E.Y. Yoon, J. Gubicza, H.S. Kim, J.L. Lábár, T. Ungár: Microstructure and hardness of copper - carbon nanotube composites consolidated by High Pressure Torsion, Mater. Sci. Eng. A 528 (2011) 4690-4695.

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[S3] P. Jenei, J. Gubicza, E.Y. Yoon, H.S. Kim, J.L. Lábár: High temperature thermal stability of pure copper and copper - carbon nanotube composites consolidated by High Pressure Torsion, Composites: Part A 51 (2013) 71–79.

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