Cambridge University Press
978-1-107-40803-6 - Mechanical Behavior at Small Scales — Experiments and Modeling: Materials Research Society Symposium Proceedings: Volume 1224
Editors: Jun Lou, Erica Lilleodden, Brad L. Boyce, Lei Lu, Peter M. Derlet, Daniel Weygand, Ju Li, Michael Uchic and Eric Le Bourhis
Excerpt
More information

Nanostructured Materials

Cambridge University Press
978-1-107-40803-6 - Mechanical Behavior at Small Scales — Experiments and Modeling: Materials Research Society Symposium Proceedings: Volume 1224
Editors: Jun Lou, Erica Lilleodden, Brad L. Boyce, Lei Lu, Peter M. Derlet, Daniel Weygand, Ju Li, Michael Uchic and Eric Le Bourhis
Excerpt
More information

Mater. Res. Soc. Symp. Proc. Vol. 1224 © 2010 Materials Research Society **1224-FF10-37**

Variable Elastic-Plastic Properties of the Grain Boundaries and Their Effect on the Macroscopic Flow Stress of Nanocrystalline Metals

Malgorzata Lewandowska , Romuald Dobosz , Krzysztof J. Kurzydlowski

Warsaw University of Technology, Faculty of Materials Science and Engineering,

Woloska 141, 02-507 Warsaw, POLAND.

ABSTRACT

The paper reports new experimental results describing properties and microstructure of nanocrystalline metals. Nano- and sub-micron aluminium has been produced by hydrostatic extrusion at ambient tempearture. The structures have been quantified in terms of size of grains and misorientation of the grain boundaries. Different average size of grains, variable normalized width of grain size distribution and changing grain boundary misorientation distribution functions have been revealed depending on processing parameters. The results of the tensile tests showed that the average grain size, grain size distribution and the distribution function of misorientation angles influence the flow stress of obtained nano-metals.

In order to explain the observed difference in the properties of nano- and micro-sized aluminium alloys, a Finite Element Method models have been developed, which assumes that both grain boundaries and grain interiors may accommodated elastic and non-linear plastic deformation. These models assumed true geometry of grains (which differed in size and shape). Also, variable mechanical properties of grain boundaries have been taken into account (elastic modulus, yield strength and work hardening rate). The results of modelling explain in a semi-quantitative way macroscopic deformation of nano-crystalline aggregates. In particular, they illustrate the importance of the interplay between properties of grain boundaries and grain interiors in elastic and plastic regime.

INTRODUCTION

A vast majority of engineering materials are used in polycrystalline form which makes grain boundaries one of the most important microstructural elements. Their role is particularly important in the case of fine grained and nanocrystalline materials as their surface area per unit volume is substantially greater than in conventional micro-structured polycrystalline counterparts. This can be supported by simple geometric considerations which indicate that for an average grain size of 10 nm the volume fraction of atoms located at grain boundaries is 25% assuming 1 nm thickness of grain boundaries. As a result, the properties of nanocrystalline materials are largely governed by grain boundaries.

The effect of the grain boundaries on flow stress of metals has been a subject of numerous experimental and theoretical studies [1-2]. It is usually described by the Hall-Petch relationship which predicts linear dependence of the flow stress on the inverse square root of grain size. Hall-Petch relationship was experimentally proven for a wide range of materials with average grain sizes ranging from several hundreds of microns to dozens of nanometres.

However, it has been found, in this context, that below a certain critical value of grain size (typically 10-30 nm) the inverse Hall-Petch relationship is observed [3].

Recently, remarkable progress in the understanding of the effect of grain boundaries has been made due to advances in modelling of plastic behaviours of nano-polycrystalline aggregates. In particular, so called composite model was successfully used to explain inverse Hall-Petch relationship [4]. In this approach, nanocrystalline material is considered as a composite consisting of non-zero-thickness grain boundaries and grain interiors, which have different mechanical properties [5]. Despite these achievements, some key questions with regard to the role of grain boundaries remain unanswered and quantification of their effect on the macroscopic properties is still a challenge.

EXPERIMENTAL

In order to experimentally evaluate the effect of grain boundaries on the macroscopic flow stress of aluminium alloys, various sub-micro grained structures were obtained in technically pure 1050 aluminium via processing by hydrostatic extrusion and subsequent low-temperature annealing at temperature ranging from 100 to 300°C. The samples were hydrostatically extruded to 3 mm wire in multistep extrusion processes starting with either 50 or 20 mm billets (a total true strain of 5.4 or 3.8, respectively). They were water cooled at the exit of the die to minimize the effect of adiabatic heating during the process. More information on the processing procedure can be found elsewhere [6].

The obtained microstructures were evaluated qualitatively by TEM observations and quantitatively using computer aided image analysis. Specimens for TEM studies were cut perpendicularly to the extrusion axis. The foils have been examined in a Jeol JEM-1200 electron microscope operated at 120 kV. The grain size was described using the equivalent grain diameter d (defined as the diameter of a circle of equal area to the surface area of a given grain) and variation coefficient CV(d) defined as a ratio of standard deviation SD(d) to the mean value. The misorientation angles were determined for a population of randomly selected grain boundaries. Crystallographic orientations of individual grains were calculated from Kikuchi lines patterns obtained in TEM by convergent beam diffraction. These patterns were subsequently used to calculate misorientations across the boundaries. For each annealing temperature, a population of at least 100 grain boundaries was analyzed.

As HE processed billets are relatively large in dimensions, it was possible to characterize the mechanical properties of the processed materials in tensile tests with relatively good statistics. Tensile tests were conducted at room temperature and at initial strain rate of 10^{-3} s^{-1}.

Finite Element Method, FEM, models, which have been developed in the present study, assume that both grain boundaries and grain interiors may accommodated elastic and non-linear plastic deformation. These models are based on true geometry of grains extracted from TEM images. The grains differed in size, size diversity and shape. Their populations in a given model were described by the average grain diameter, E(d) and coefficient of variation, CV(d). Also, variable mechanical properties of grain boundaries have been taken into account (i.e. elastic modulus, yield strength and work hardening rate). For FEM calculations, mechanical properties of grain interiors were kept constant whereas mechanical properties of grain boundaries were systematically varied to evaluate their effect on the macroscopic reaction of the polycrystalline aggregates to the applied force in a tensile test.

RESULTS

Hydrostatic extrusion results in a significant grain refinement in metals as was already confirmed for a number of alloys, e.g. aluminium [7,8], titanium [9], steels [10,11]. In the case of technically pure aluminium, the HE induced microstructures consist of fairly equiaxial, well developed grains almost free of dislocations, as illustrated in figure 1. The grains are slightly elongated in the direction parallel to extrusion direction (figure 1c). However, it should be noted that depending on processing parameters (applied strain and annealing conditions), the microstructures differ in the size of grains which usually is described using an average value.

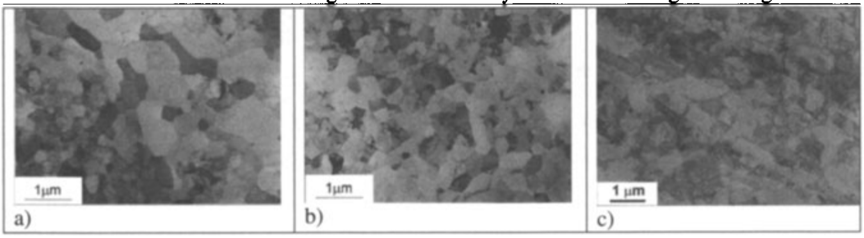

Figure 1. Typical TEM micrographs of 1050 aluminium processed by hydrostatic extrusion with a true strain of 2.7(a) and 3.8 (b,c). (a,b) transverse section, (c) longitudinal section.

In this context, it should be noted that the use of an average value of grain size is fully justified only under assumption that the size of grains can be described by the same size distribution function. On the other hand, polycrystalline materials must be viewed as stochastic populations of grains which may differ significantly in size and shape. As a result, the same average value may ascribe for far different grain structures, as illustrated in figure 1. In addition, individual grain boundaries may have different properties due to different misorientation angles. The results of measurements of various microstructural parameters (namely average grain size, grain size coefficient of variation and distribution of misorientation angles) for the samples investigated in this study are listed in Table I.

Table I. Microstructural parameters of 1050 aluminium after various processing conditions

spl	processing parameters	YS [MPa]	ε_t [%]	grain size		fraction of grain boundaries [%]		
				E(d) [nm]	CV(d)	$\theta<5^{\circ}$	$5^{\circ}<\theta<15^{\circ}$	$\theta>15^{\circ}$
1	as-received	149	7.1	920	0.41	-	-	-
2	HE, $\varepsilon=1.4$	176	6.3	610	0.43	65	25	10
3	HE, $\varepsilon=2.7$	175	8.1	580	0.42	22	33	45
4	HE, $\varepsilon=3.8$	202	6.5	600	0.36	20	20	60
5	HE, $\varepsilon=5.4$	205	2.8	320	0.40	8	20	72
6	HE, $\varepsilon=5.4$ + 200°C for 1 h	167	3.3	450	0.50	7	15	77
7	HE, $\varepsilon=5.4$ + 300°C for 3 hrs	53	26.9	35,000	0.45	-	-	-

It should be further noted that grain structures produced by hydrostatic extrusion, alone or in combination with annealing, differ significantly not only in grain size but also in grain size diversity (the variation coefficients of grain size vary from 0.36 to 0.50) and the distribution of misorientation angles of the grain boundaries.

The experimental values of yield strength for sub-micro grained 1050 aluminium plotted in the coordinates of Hall-Petch equation (against inverse square root of the average grain size), show a significant scattering from the linear dependence, as presented in figure 2. For the same average grain size, the samples exhibit far different properties or the other way round the same yield strength is observed for far different grain sizes. The in-depth analysis of microstructural parameters, shown in Table I, indicates that grain size diversity expressed by variation coefficient significantly influences the flow stress, as illustrated in figure 2. The Hall-Petch line is drawn for the data points obtained for samples with CV~0.41. It can be noted that the flow stress for CV~0.50 diverts from the Hall-Petch line downwards. On the other hand, the flow stress for CV=0.36 is shifted upwards. The character of grain boundaries plays also a significant role.

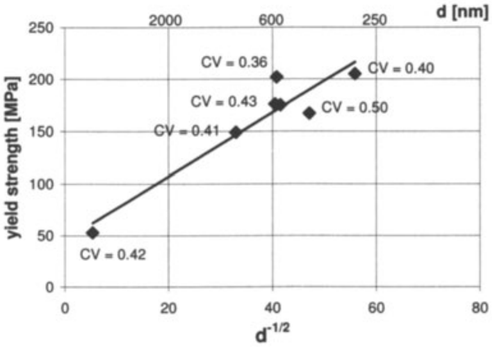

Figure 2. Plot of Hall-Petch relationship for 1050 aluminium processed by hydrostatic extrusion and subsequent annealing

In order to have a better insight into the role of grain boundaries on flow stress of nanocrystalline materials, a comprehensive FEM model (figure 3a) has been developed which takes into account such features of nano-polycrystalline metals as: (1) variable geometry of grains; (2) varied mechanical properties (elastic modulus, yield stress and work hardening rate) of grain boundaries and (3) grain size dependant flow stress of grain interiors. The detail model description can be found elsewhere [12].

FEM computations for different combination of the properties of grain boundaries have been performed to obtain macroscopic stress-strain curves (the flow stress of nano-polycrystalline aggregates was normalized to the flow stress of micro-grained counterpart), as exemplified by the ones presented in figure 3b. The computed changes in the macroscopic Young modulus and flow stress of nano-polycrystalline aggregate as a function of varying grain boundary Young modulus (E^B) normalized to the Young modulus of the grain interior (E^I) are shown in figure 4. It can be noted that the effect of grain boundary Young modulus is particularly pronounced when grain boundaries are less stiff than grain interiors. In such a case, a substantial decrease in Young modulus and macroscopic flow stress of nano-polycrystalline aggregate is observed.

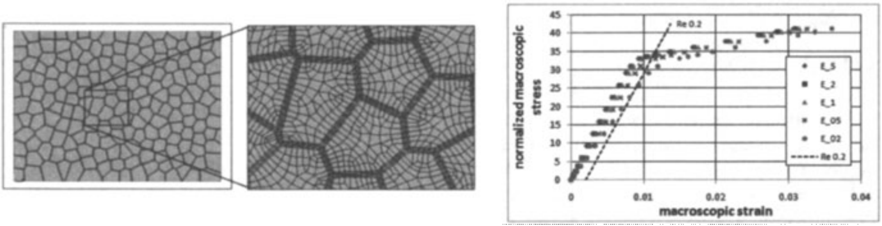

Figure 3. FEM model of nano-polycrystalline metals (a) and macroscopic stress-strain curves of

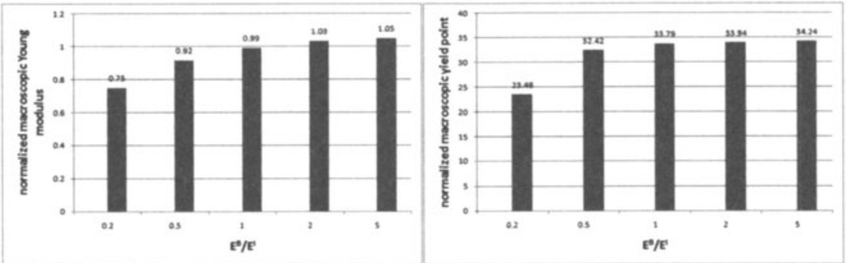

Figure 4. The effect of varying value of grain boundary Young modulus, E^B, normalized to the Young modulus of the grain interior, E^I on (a) Young modulus and (b) yield point of nano-polycrystalline aggregate

Young modulus of the grain interior, E^I on (a) Young modulus and (b) yield point of nano-polycrystalline aggregate

Figure 5 illustrates the effect of grain boundary yield strength R_e^G (the same in normal and in-plane directions) on macroscopic Young modulus, strain hardening rate and yield strength of nano-polycrystalline aggregate. A profound reduction in macroscopic Young modulus and yield strength is observed for low values of grain boundary strength.

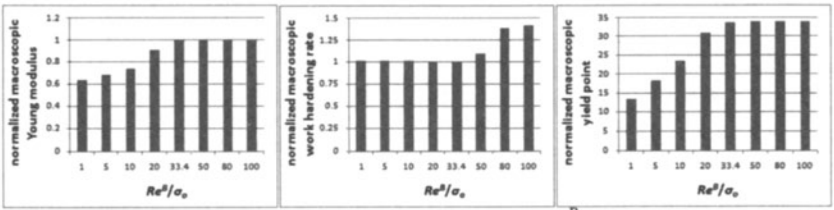

Figure 5. The effect of varying grain boundary flow stress, R_e^B, normalized to the flow stress of the monocrystal of analyzed material, σ_o on: (a) Young modulus, (b) work hardening rate and (c) yield point of nano-polycrystalline aggregate

Cambridge University Press
978-1-107-40803-6 - Mechanical Behavior at Small Scales — Experiments and Modeling: Materials Research Society Symposium Proceedings: Volume 1224
Editors: Jun Lou, Erica Lilleodden, Brad L. Boyce, Lei Lu, Peter M. Derlet, Daniel Weygand, Ju Li, Michael Uchic and Eric Le Bourhis
Excerpt
More information

The above described findings have implications to understanding the mechanics of nano-metals. It is shown that low macroscopic Young modulus and yield strength in nano-metals might be heavily dependent on the mechanical properties of grain boundaries. This is due to the topology of grain boundaries which in a polycrystalline aggregate form a percolated structure.

CONCLUSIONS

It is shown here that macroscopic tensile behaviours of fine grained polycrystals depend on the average grain size, grain size distribution and the distribution function of misorientation angles. Experimental and numerical modeling results also reveal a significant impact of grain boundary properties on macroscopic deformation of nano-crystalline aggregates. In particular, they illustrate the importance of the interplay between properties of grain boundaries and grain interiors in elastic and plastic regime.

ACKNOWLEDGMENTS

This work was carried out within a NANOMET Project financed under the European Funds for Regional Development (Contract No. POIG.01.03.01-00-015/08). Hydrostatic extrusion experiments were carried out at the Institute of High Pressure Physics of Polish Academy of Sciences within the project coordinated by the Faculty of Materials Science and Engineering of Warsaw University of Technology.

REFERENCES

1. N. Hansen, Mat. Sci. Eng. A409, 39-45 (2005)
2. K.J. Kurzydlowski, B. Ralph, J.J. Bucki, A. Garbacz, *Mater. Sci. Eng.* **A205**, 127-132 (1996)
3. J. Schiotz, F.D. di Tolla, K.W. Jacobsen, *Nature* **391**, 561-563 (1998)
4. R. A. Masumura, P. M. Hazzledine and C. S. Pande, *Acta mater.* **46**, 4527-4534 (1998)
5. X. S. Kim, Y. Estrin, *Acta mater.* **53**, 765-772 (2004)
6. M. Lewandowska, K.J. Kurzydlowski, *J. Mat. Sci.* **43,** 7299-7306 (2008)
7. K. Wawer, M. Lewandowska, K.J. Kurzydlowski, *Mat. Sci. Forum* **584-586,** 541-54 (2008)
8. M. Lewandowska, W Pachla, K.J. Kurzydlowski, *Int. J. Mat. Res. (formely Z.Metallkd)* **98**, 172-177 (2007)
9. H. Garbacz, M. Lewandowska, W. Pachla, K.J. Kurzydlowski, *J Microscopy* **223**, 272-274 (2006)
10. M. Lewandowska, A.T. Krawczynska, M. Kulczyk, K.J. Kurzydlowski, *J. Nucl. Mat.* **386-388**, 499-502 (2009)
11. A.T. Krawczynska, M. Lewandowska, K.J. Kurzydlowski, *Solid State Phenomena* **140**, 173-178 (2008)
12. R. Dobosz, T. Wejrzanowski, K. J. Kurzydlowski, *Computer Methods in Material Science* **9**, No. 1, (2009)

Cambridge University Press
978-1-107-40803-6 - Mechanical Behavior at Small Scales — Experiments and Modeling: Materials Research Society Symposium Proceedings: Volume 1224
Editors: Jun Lou, Erica Lilleodden, Brad L. Boyce, Lei Lu, Peter M. Derlet, Daniel Weygand, Ju Li, Michael Uchic and Eric Le Bourhis
Excerpt
More information

Mater. Res. Soc. Symp. Proc. Vol. 1224 © 2010 Materials Research Society **1224-FF10-32**

Deformation behavior of nanocrystalline Co-Cu alloys

Motohiro Yuasa[1], Hiromi Nakano[2], Kota Kajikawa[1], Takumi Nakazawa[1], Mamoru Mabuchi[1]

[1] Graduate School of Energy Science, Kyoto University, Yoshidahonmachi, Sakyo-ku, Kyoto, 606-8501, Japan
[2] Cooperative Research Facility Center, Toyohashi University of Technology, 1-1 Hibarigaoka, Tempaku-cho, Toyohashi 440-8580, Japan

ABSTRACT

Three kinds of nanocrystalline Co-Cu alloys: a nanocrystalline Co-Cu alloy with nanoscale lamellar structure, a supersaturated solid solution Co-Cu alloy and a nanocrystalline two-phase Co-Cu alloy were processed by electrodeposition, and their mechanical properties were investigated at room temperature. These nanocrystalline Co-Cu alloys showed the high hardness and the low activation volume. The mechanical properties of the nanocrystalline Co-Cu alloys strongly depended on the grain boundary characteristics. Molecular dynamics simulations were performed in the two-phase nanocrystalline Co-Cu alloy to investigate the dislocation emission at the Co/Cu interface. The MD simulations showed that the stacking faults, which are generated by the intense geometrical strain at the Co/Cu interface, play an important role in the dislocation emission.

INTRODUCTION

Co alloys are one of promising metallic materials because they exhibit high heat resistance, ferromagnetism and so on. For various applications, it is desirable to improve the mechanical properties of Co alloys. Nanocrystallization can give rise to a significant enhancement of mechanical properties in metallic materials. However, nanocrystalline metals tend to be very brittle with a ductility of less than a few percent in tensile tests [1,2], due to the absence of dislocation activity [3]. It is accepted that nanocrystalline metals show the high hardness (high strength) and the low activation volume [4-9]. These features of nanocrystalline metals are attributed to emission of dislocations at the grain boundaries, and the grain boundaries play a critical role in deformation of nanocrystalline metals. Hence, it is required to develop nanocrystalline Co alloys with unique grain boundaries for enhancement of the mechanical properties.

In the present work, three kinds of nanocrystalline Co-Cu alloys are processed by electrodeposition, and their mechanical properties are investigated at room temperature. In addition, molecular dynamics (MD) simulations are performed in the nanocrystalline two-phase Co-Cu alloy to investigate the dislocation emission at the Co/Cu interface.

EXPERIMENTAL

Three kinds of nanocrystalline Co-Cu alloys, that is, a nanocrystalline Co-Cu alloy with nanoscale lamellar structure, a supersaturated solid solution Co-Cu alloy and a nanocrystalline two-phase Co-Cu alloy were processed by electrodeposition [10]. The electrolyte composition was $CoSO_4 \cdot 7H_2O$ (1 M) and $CuSO_4 \cdot 5H_2O$ (0.025 M). Microstructure of the Co-Cu alloys was investigated by transmission electron microscopy. Mechanical properties of the Co-Cu alloy were investigated by the hardness and tensile tests at room temperature. The hardness tests were performed with a diamond Berkovich tip at constant loading rates of 13.24, 1.324 and 0.378 mN/s.

RESULTS AND DISCUSSION

Nanolamellar Co-Cu alloy

A transmission electron microscopy image of a nanocrystalline Co-Cu alloy with nanoscale lamellar structure is shown in Fig. 1. The grain size of the Co-Cu alloy was 110 nm. Note that most of the grains contained a high-density fine nanoscale lamellar structure. In previous studies [11,12], the nanocrystalline Cu with nanoscale twins with a spacing of tens of nanometers was fabricated by electrodeposition. On the other hand, the Co-Cu alloy developed in the present work contained nanoscale lamellar structure with a much smaller spacing of 3 nm.

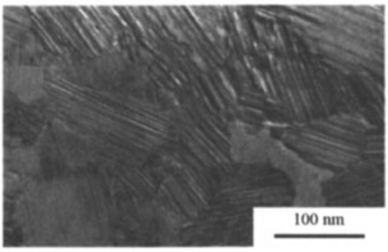

Figure 1. A transmission electron microscopy image of the nanocrystalline Co-Cu alloy with nanoscale lamellar structure.

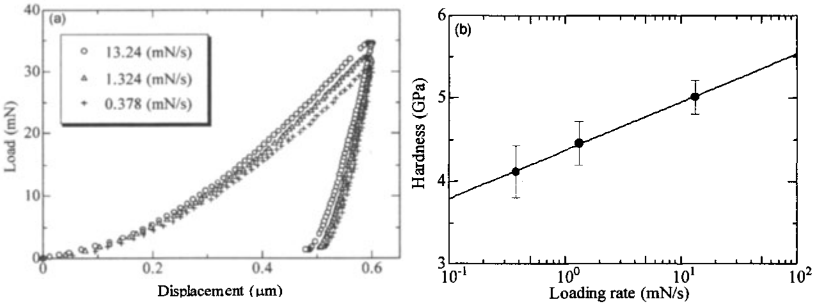

Figure 2. The results of hardness tests for the nanocrystalline Co-Cu alloy with nanoscale lamellar structure, (a) load-displacement curves at three different loading rates and (b) variation in hardness as a function of loading rate.