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High strength–high conductivity nanostructured copper wires prepared by spark plasma sintering and room-temperature severe plastic deformation

Claire Arnaud^{a,b}, Florence Lecouturier^b, David Mesguich^a, Nelson Ferreira^b,
Geoffroy Chevallier^{a,c}, Claude Estournès^{a,c}, Alicia Weibel^a, Alain Peigney^a,
Christophe Laurent^{a,*}

^a Université de Toulouse, Institut Carnot CIRIMAT, UMR CNRS-UPS-INP 5085, Université Paul-Sabatier, 118 Route de Narbonne, F-31062 Toulouse Cedex 9, France
^b Laboratoire National des Champs Magnétiques Intenses, UPR CNRS-UPS-INSA-UJF 3228, 143 Avenue de Rangueil, F-31400 Toulouse, France
^c Plateforme Nationale CNRS de Frittage Flash, PNF2 MHT, Université Paul-Sabatier, 118 Route de Narbonne, F-31062 Toulouse Cedex 9, France

A B S T R A C T

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Mechanical strength
Electrical resistivity

A pure copper cylinder with micrometric grains was prepared by spark plasma sintering and was wire-drawn at room temperature. The ultimate tensile strength of the conducting wires is 600 MPa at room temperature. This originates from the propagation of dislocations by an Orowan mechanism in grains smaller than 250 nm.

1. Introduction

There is a demand for stronger yet lighter conducting copper wires, in global fields where weight is at a premium such as aeronautics, space and power transportation as well as in niche applications such as materials for high-field magnets. However, high mechanical strength and high electrical conductivity seem to be mutually exclusive properties of metals. In polycrystalline, ‘engineering’ metals, the strength is limited by the onset of plastic deformation, which is mainly carried by lattice dislocations within the crystalline grains. To strengthen a metal, this dislocation motion has to be reduced. This can be achieved by alloying, by introducing another phase or by introducing extra grain boundaries through grain refinement. Strengthening by nanostructuring [1–8] is highly attractive as compared to alloying, because it requires little extra energy and allows for easy recycling of the metal. In general, all these strengthening methods also increase scattering of the conducting electrons, i.e. they decrease the electrical

conductivity. This impasse was broken when it was shown that a specific type of grain boundary, the coherent twin boundary, can strengthen electrochemically deposited copper films without introducing additional electron scattering, resulting in high strength–high conductivity thin films [9–11]. Similar results were achieved with nanotwinned bulk materials prepared using cryo-deformation [12–14]. However, a considerably altered electrical conductivity suggested the formation of non-coherent twin boundaries or destructed twin orientation relationship in these cryo-drawn wires [14]. Interestingly, these authors observed that a coarse-grained initial microstructure yields a slightly lower mechanical strength for wires drawn at room temperature while the ultimate tensile strength of cryo-drawn copper is not altered by the initial microstructure. Indeed, the tensile strength of a Cu wire deep-drawn at room temperature from an initial Cu cylinder with a mean grain size of 9.4 μm reached 460 MPa [15] and is usually below 400 MPa [1]. Therefore, it was hypothesized for the present work that, in order to produce high-strength wires by room-temperature wire-drawing, it is a necessity to start from a precursor cylinder with grains one order of magnitude smaller, i.e. micrometer-sized, than conventional cylinders. The modest degree of grain refinement thus endured would not introduce much

* Corresponding author.

E-mail address: laurent@chimie.ups-tlse.fr (C. Laurent).

additional scattering, maintaining a high electrical conductivity. However, producing such a micrometer-sized Cu precursor cylinder is not straightforward because of the rapid increase in grain size upon exposition to the elevated temperatures necessary for sintering a Cu powder. To circumvent this, we propose to prepare the precursor cylinder with spark plasma sintering (SPS), the application of a pulsed direct current to a pressed powder [16,17]. The advantages of SPS over other sintering methods include lower sintering temperatures and shorter sintering times, which can produce Cu pellets with limited grain growth [18–20]. Cu wires then produced by room-temperature wire-drawing show an electrical conductivity of more than 84% International Annealed Copper Standard (IACS), at room temperature with an ultimate tensile strength (UTS) of 600 MPa, 50% higher than when using standard precursor cylinders.

2. Experimental procedures

A Cu cylinder was prepared by SPS (see supporting information). It was cold-drawn at room temperature. Samples of wires were typically 70 cm or 150 cm long. The total true strain (η) reached 7.29 with η given by $\eta = \ln(A_0/A)$ where A_0 is the cross-sectional area of the cylinder and A is the cross-sectional area of the wire. For the sake of comparison, wires were also prepared by the same route using a conventional cylinder (average grain size of the order of 10 μm) prepared from standard cast oxygen-free high conductivity (OFHC) Cu.

Analysis of the X-ray diffraction (XRD) patterns (see supporting information) revealed only traces of Cu_2O for the powder while no copper oxide is detected after SPS (Fig. S1), reflecting that the SPS experimental conditions are reducing. It is known [21] that for Cu nanopowders, the initial presence of CuO will favor the formation of Cu_2O during SPS due to the anti-dismutation reaction $\text{Cu} + \text{CuO} \rightarrow \text{Cu}_2\text{O}$, but it is not the case here.

The relative density of the cylinder and wires was measured by Archimedes' method. The cylinder and transversal and longitudinal sections of selected wires were observed by scanning electron microscopy (SEM, JEOL JSM 6700 F) and transmission electron microscopy (TEM, JEOL JEM 2100 F operated at 120 kV). The electrical resistivity was measured at 293 K and 77 K for wires W5–W12 using the four probe method with a maximum current of 100 mA to avoid heating the wires. Microhardness was determined from indentation tests (1 N for 10 s in air at room temperature) performed on the polished transverse surface of wires by loading with a Vickers indenter (Shimadzu HMV M3). Tensile tests (INSTRON 1195 machine) were performed at 293 K and 77 K on 170 mm long specimens. The tensile direction was parallel to the wire-drawing direction. During the tests, precise stresses were measured by the stress gauge system (250 N), under displacement control at a speed rate of $1.6 \times 10^{-5} \text{ m s}^{-1}$. The denser wires were tested.

3. Results and discussion

3.1. Density and microstructure of the cylinder and wires

The relative density for the cylinder is equal to $86 \pm 1\%$. While not high, this value was found convenient for the rest of the study, in particular because a too high density hampers the deformability of the cylinder during wire-drawing, resulting in sample breaking. TEM observations (Fig. 1) of a transversal section of the cylinder reveal that the Cu grains have not grown significantly from the original size ($1.0 \pm 0.5 \mu\text{m}$) and contain twins *ca.* 100 nm thick (arrowed in Fig. 1) with a spacing about 300 nm. Zhang et al. [22]

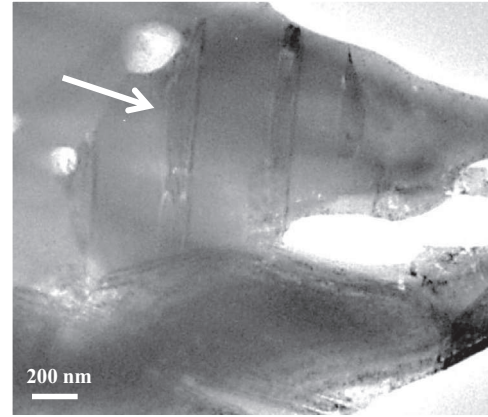


Fig. 1. TEM bright-field image of the transversal section of the Cu cylinder (8 mm in diameter) prepared by SPS showing a micrometric grain containing twins (arrowed).

observed the presence of extensive nanoscale twins with different spacing (about 60 nm and less than 2 nm) in bulk Cu prepared by SPS in different conditions than the present ones (300 °C, 600 MPa). They observed that the average nanoscale twin width increases upon the increase of the sintering temperature. Thus, the twins observed in the present sample could correspond to their larger-spacing twins whereas no twins with very small spacing are observed because we use a much lower SPS uniaxial pressure (25 vs 600 MPa).

Twelve wires (designated W1–W12 hereafter) were selected for the study (Table 1). For example, the diameter of W8 and W12 is equal to 0.506 and 0.198 mm, respectively and η is equal to 5.41 and 7.29, respectively, corresponding to very large strains [23]. The relative density is significantly higher for W1 ($94 \pm 1\%$) than for the cylinder ($86 \pm 1\%$) and increases up to $97 \pm 3\%$ for W8. It is probably still higher for W9–W12 but the measurement uncertainty is too high to give a meaningful value. The fact that the wires did not break during the wire-drawing (WD) process reveals that the residual porosity in the cylinder allows for grain rotation in the beginning of the drawing process (see XRD section in supporting information) and, once a higher density is achieved, for grain deformation.

Transverse SEM (Fig. 2a) and TEM observations (Fig. 2c) of W8 reveal 50–400 nm grains, three quarter of them smaller than 260 nm (Fig. 2b). TEM observations of a longitudinal section of W8 (Fig. 3), i.e. parallel to the WD direction, show the so-called lamellar microstructure [23]. Grains are dramatically elongated over

Table 1

Diameter, electrical resistivity and microhardness of the cylinder and wires. Sample, diameter, the corresponding total true strain η , electrical resistivity at 293 K ($\rho_{293 \text{ K}}$) and 77 K ($\rho_{77 \text{ K}}$), Vickers microhardness (Hv).

Sample	Diameter (mm)	η	$\rho_{293 \text{ K}}$ ($10^{-8} \Omega \text{ m}$)	$\rho_{77 \text{ K}}$ ($10^{-8} \Omega \text{ m}$)	Hv (GPa)
Cylinder	7.560	0.00	–	–	0.58
W1	3.300	1.66	–	–	1.21
W2	2.064	2.60	–	–	1.23
W3	1.511	3.23	–	–	1.26
W4	0.809	4.47	–	–	1.33
W5	0.640	4.94	2.07	0.48	1.43
W6	0.592	5.10	2.01	0.47	1.44
W7	0.547	5.26	2.05	0.48	1.45
W8	0.506	5.41	2.05	0.48	1.48
W9	0.401	5.88	2.08	0.49	1.39
W10	0.293	6.51	2.19	0.52	1.52
W11	0.251	6.82	2.16	0.52	1.54
W12	0.198	7.29	2.24	0.55	1.61

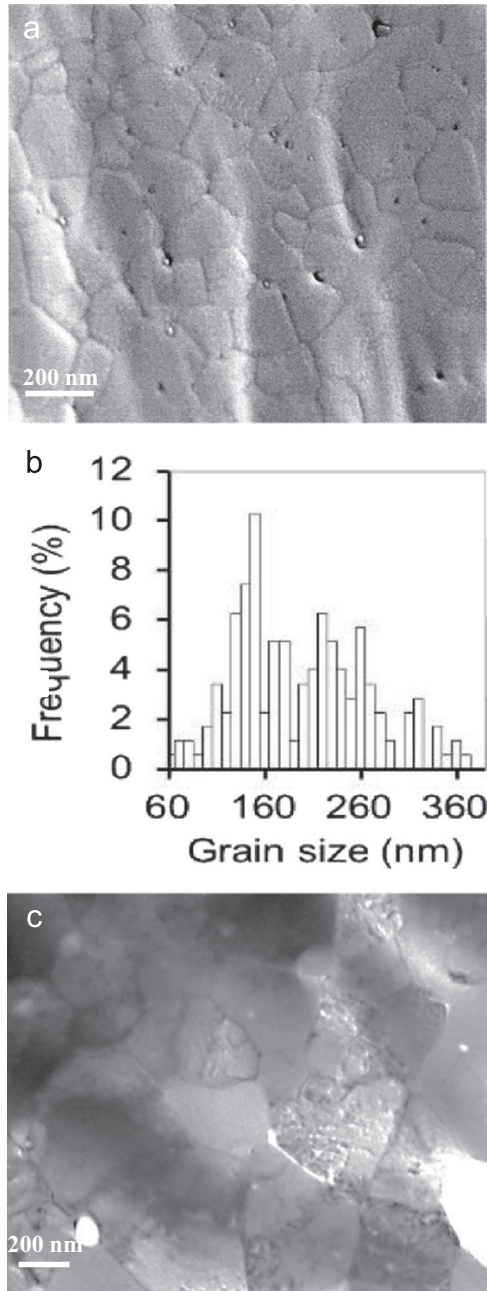


Fig. 2. (a) SEM image of the transversal section area of wire W8 (0.506 mm in diameter); (b) grain diameter distribution calculated from similar images, (c) TEM bright-field image of the transversal sections.

10 μm and the length/diameter ratio is in the range 25–100. Dislocation substructures are observed within the lamellas (arrowed in Fig. 3b). Interestingly, no twins were observed, in contrast to what was observed within the grains for the SPS cylinder (Fig. 1). It is proposed that the deformation during WD provoked their migration due to twin boundaries acting as non-regenerative dislocations sources [24]. Interestingly, coherent twin boundaries were not observed, in contrast to results reported for cryo-drawn wires [12–14].

3.2. Electrical and mechanical properties

For W5–W12, the electrical resistivity is in the range $(2.07\text{--}2.24) \cdot 10^{-8} \Omega \text{ m}$ at 293 K and in the range $(0.48\text{--}0.55) \cdot 10^{-8} \Omega \text{ m}$ at 77 K (Table 1). The 77 K values are lower because the electron–

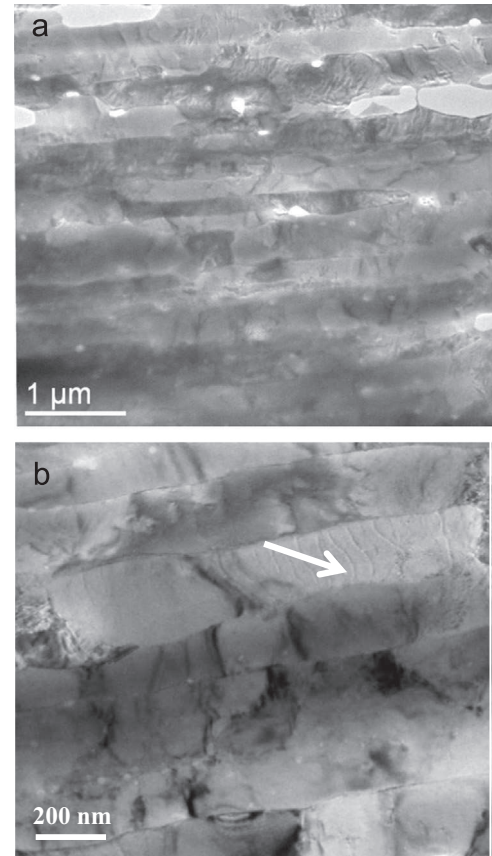


Fig. 3. (a) TEM bright-field image of the longitudinal section of wire W8 (0.506 mm in diameter); (b) higher magnification image; dislocation substructures within a Cu grain are arrowed.

phonon interactions are negligible and the residual resistivity is only controlled by the electron-defect interactions. For both measurement temperatures, there is a slight increase in resistivity upon the decrease in wire diameter and thus upon the increase in η . This reflects that the progressive deformation provoked an increase in the density of defects such as grain boundaries, sub-grains and dislocation loops acting as scattering centers for conduction electrons. Moreover, the size range achieved (most grains smaller than 260 nm for W8 for example) is probably close to the electron mean-free-path, at least at 77 K, which will also contribute to a higher resistivity. For W8, the resistivity at 293 K ($2.05 \cdot 10^{-8} \Omega \text{ m}$) corresponds to an electrical conductivity of 84.1% IACS, slightly lower than for same-diameter wires prepared from conventional cylinders: 93.0% IACS (this work) and 96.5% IACS [15]. This conductivity loss is only moderate if one hypothesizes that the copper grains for W8 are about one order of magnitude smaller than for the conventional wires, as is the case for the corresponding cylinders. This homothetic size reduction upon the deformation still has to be verified but is nevertheless considered to be reasonable.

The Vickers microhardness of the cylinder is equal to $0.58 \pm 0.01 \text{ GPa}$ (Table 1) and the value is at least doubled for all tested wires, increasing from 1.21 to 1.48 GPa upon the decrease in diameter, which could reflect strain hardening and/or grain refinement. For wires prepared using the conventional cylinder, the microhardness is constant (1.25 GPa) regardless of diameter.

Typical stress–strain curves are shown in Fig. 4. Strain was determined from crosshead displacement, without any correction of machine rigidity. Thus, the wide observed elastic domain is an artefact due to the experimental set-up. Ductile fracture was observed for all wires submitted to the tensile test. The ultimate

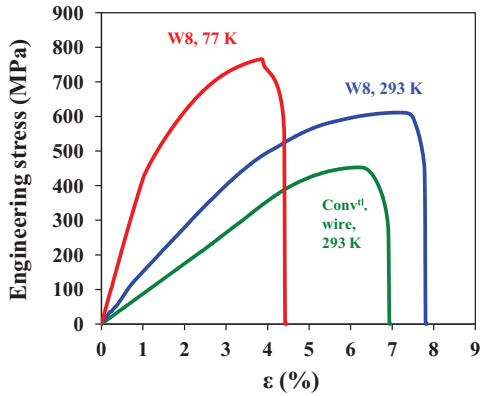


Fig. 4. Stress–strain curves at 293 and 77 K for wire W8 (0.506 mm in diameter) prepared from the SPS cylinder and at 293 K for the corresponding conventional wire.

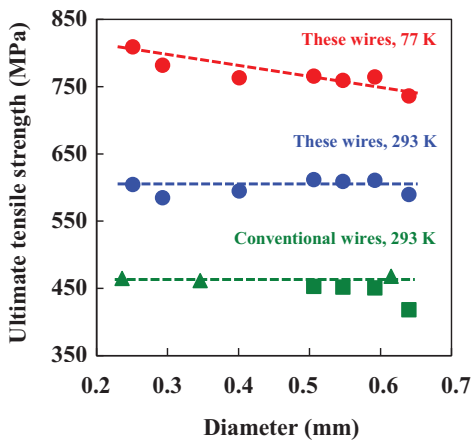


Fig. 5. Ultimate tensile strength vs diameter for wires W5–W11 prepared from the SPS cylinder (“these wires”), at 77 K (●) and 293 K (●). Data (293 K) for wires prepared using a conventional cylinder: this work (■) and ref.[15] (▲). The dashed lines are guides to the eye.

tensile strength (UTS) at 293 K (Fig. 5) for the present conventional wires (450 MPa) is in good agreement with the reported value (460 MPa) for a Cu wire deep-drawn at room temperature using a Cu cylinder with a mean grain size of 9.4 μm [15]. This indicates that both our WD and measurement processes are valid. Although there is no clear transition between the elastic and the plastic domains, it was determined that the yield stress at 293 K is equal to 590 MPa, close to the UTS at 293 K equal to about 610 MPa. This value is significantly higher than those for pure Cu conventional wires (450–460 MPa). We compare pure Cu wires and therefore differences in properties arise from differences in microstructure, in particular the width and length of the lamellas. The combination of SPS, for the production of a Cu cylinder with micrometer-sized grain, and room-temperature wire-drawing, allowed us to achieve the preparation of wires with a finer microstructure and therefore showing a much higher strength. To the best of our knowledge, such high UTS values have never been reported before for bulk pure Cu wires prepared with only two steps, SPS and room-temperature drawing, without any annealing applied. Moreover, the UTS at 77 K increases upon the decrease in diameter (Fig. 5) in the range 736–809 MPa, thus about 40% higher than for our conventional wire, *ca.* 550 MPa. Thus, the present wires could be used under cryogenic conditions, which is interesting for applications in non-destructive pulsed magnets operating in liquid nitrogen (77 K) [25], for example.

Interestingly, the present high UTS values are obtained whereas

no coherent twin boundaries are observed within the elongated grains of the wires (Fig. 3). The plasticity mechanisms during WD are assumed to be based on Orowan-type dislocation glide, a single-loop mechanism that promotes high strength [26–29]. Note that the Orowan mechanism (i.e. propagation of single dislocation loops), is not limited to cases where precipitates or dispersoids are present, but is also operative for fine scale lamellar structures [26], metallic multilayered composites [27], multi-filamentary nanostructured composites [28] and ultrafine grained fcc metals [29], the latter example being close to the present case. The mechanical strength of wire W8 was modeled, taking into account the contribution of both the cold-working and the strengthening due to the Orowan-type grain diameter dependence [26]. The yield stress (σ) was calculated using $\sigma = \sigma_{\text{cold-worked copper}} + \sigma_{\text{Orowan}}$, assuming $\sigma_{\text{cold-worked copper}} = 450$ MPa [5] and $\sigma_{\text{Orowan}} = M(1.2 \mu\text{b}/2\pi d) \ln(d/b)$, where M is the Taylor factor ($M = 3.16$), μ the shear modulus ($\mu = 42.5$ GPa), b the length of the Cu $\langle 111 \rangle$ Burgers vector ($b = 0.256$ nm) and d the grain diameter (lamella width). In order to take into account the grain diameter distribution (Fig. 2b), two populations were considered, 150 nm (40 vol%) and 250 nm (60 vol%). The calculation gives $\sigma = 670$ MPa. Taking into account the relative density, this value is adjusted to 650 ± 20 MPa, which indeed is in good agreement with the measured yield stress (590 MPa).

4. Conclusions

Pure copper wires with a very high tensile strength, high yield stress and high electrical conductivity have been produced by room-temperature wire-drawing. This was made possible by using a pure copper cylinder prepared by spark plasma sintering, as a precursor with micrometer-sized grains, 10 times smaller than for conventional cylinders. This innovative approach minimizes the degree of grain refinement necessary to produce wires with very small grains and thus minimizes the formation of defects. No coherent twin boundaries were observed within the elongated grains of the wires and the very high ultimate tensile strength of the wires (over 600 MPa at room temperature and as high as 800 MPa at 77 K) was shown to originate from the propagation of dislocations by an Orowan-type dislocation glide mechanism in grains smaller than 250 nm. It is true that the additional step of spark plasma sintering is not a minor modification to the general method, but spark plasma sintering can be up-scaled, and also it obviates the need for cryo-deformation. Thus, in our view, our proposed route may be worth it for wires used in scientific research and practical utilities in fields requiring ultra-strong yet much lighter conducting wires, such as aerial power lines, aeronautics and space. Future works include the study of the deformation process and texture analysis of the wires by EBSD and the increase of the densification to increase electrical conductivity.

Prime novelty statement

We show that it is possible to prepare pure Cu wires, showing both a high electrical conductivity and ultimate tensile strength, by room-temperature wire-drawing, as opposed to cryo-deformation. To achieve these results, we use as the starting material for wire-drawing a Cu cylinder prepared by spark plasma sintering with micrometer-sized grains, i.e. 10 times smaller than for conventional cylinders. Thus, wires with elongated nanoscale grains are obtained with only a low degree of grain refinement and the associated formation of defects that would otherwise decrease the conductivity. We establish that the high ultimate tensile strength originates from an Orowan-type dislocation glide mechanism in

grains smaller than 250 nm.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at <http://dx.doi.org/10.1016/j.jmse.2015.09.122>.

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