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Thermo-mechanical behaviour of nanostructured copper

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Abstract

Strain rate sensitivity is measured using jump test, with respect to strain rate and temperature on nanostructured copper. The nanometal, prepared by powder metallurgy technique, is tested at room temperature over the range $1 \times 10^{-5}\,s^{-1}$ to $1.8 \times 10^{-2}\,s^{-1}$, and at moderate temperatures, between 353 K and 393 K (homologous temperature T/T_m: 0.26–0.29), over the range $1 \times 10^{-5}\,s^{-1}$ to $9 \times 10^{-3}\,s^{-1}$. Results are discussed in terms of potential ductility and forming ability.

Keywords: Nanostructured materials; Metals and alloys; Powder metallurgy; Mechanical properties

1. Introduction

Copper with grain size comprised between around 150 nm and 30 nm exhibits different mechanical behaviour than coarse-grained counterpart [1,2]. In this domain, nanometals still contain lattice dislocations; they are featured by a high strength predicted by the Hall–Petch law, and low ductility when tested at moderate strain rate ($\dot{\varepsilon} \approx 10^{-4}$ to $10^{-1}\,s^{-1}$) [3,4]. Elongation to failure drops to about 5% for copper with grain size of less than 100 nm. Intrinsically, pure nanocrystalline metals with grains lower than around 150 nm should possess some ductility at room temperature. Compared to micron-sized metals, nanometals are strain-rate sensitive with a dependence with grain size and applied strain rate. Experiments show strain-rate sensitivity (defined as $m = (\mathrm{d}\ln\sigma/\mathrm{d}\ln\dot{\varepsilon})_\varepsilon$ with σ, the true stress and ε, the true strain) for SPD copper (grain size 300 nm), $m = 0.025$ at $\dot{\varepsilon} = 6 \times 10^{-7}\,s^{-1}$ decreasing to $m = 0.010$ at $\dot{\varepsilon} = 1 \times 10^{-4}\,s^{-1}$ [5], $m = 0.026$ for consolidated ball-milled copper (grain size 60 nm) tested at $\dot{\varepsilon} = 1 \times 10^{-4}\,s^{-1}$ [6]. Similar trend was observed for other types of preparation such as electro-deposition of nCu [7] and other metals, for example SPD aluminum [8]. When m is increased, failure of the sample is delayed in connection to Hart instability criterion [9]: $1/\sigma(\mathrm{d}\sigma/\mathrm{d}\varepsilon)_\varepsilon - 1 + m \leq 0$. Empirically, Woodford [10] showed for various alloys, a quantitative correlation as an increase in ductility with increase in strain-rate sensitivity.

In this paper, we report on the measurements of the strain-rate sensitivity m as a function of $\dot{\varepsilon}$ and the temperature T, first reported by Wang et al. [11]. Experiments are carried out on pure nanostructured copper prepared by powder metallurgy having a relative density of about 98.5% and an average grain size of 90 nm [12,13]. This nanometal exhibits near-perfect elasto-plastic behaviour during tensile test carried out at $\dot{\varepsilon} = 5 \times 10^{-6}\,s^{-1}$ [14]. The deformation attained 12% with no work hardening and no apparent necking suggesting accordingly that strain-rate sensitivity is high at room temperature. From these measurements, the aim is two-fold; on the one hand, forming ability is evaluated through the measurements of m as a function of $\dot{\varepsilon}$ and T. On the other hand, the measurements of m and derived thermodynamical data inform on the deformation mechanism, which is essential to design relevant nano-architecture in terms of nanostructure and chemistry with controlled properties.

2. Measurement of the strain rate sensitivity

Velocity jump test technique was used to measure m (see for example: [15]). Experiments were carried out in compression using a MTS 20/M machine interfaced with the MTS "Testworks 4" software. MoS_2 was spread on the plates for lubrication. The tests were performed at room temperature over the strain rate range of $1 \times 10^{-5}\,s^{-1}$ to $1 \times 10^{-4}\,s^{-1}$ and over the range of $6 \times 10^{-4}\,s^{-1}$ to $1.8 \times 10^{-2}\,s^{-1}$. At moderate temperatures, tests were carried out between 353 K and 393 K

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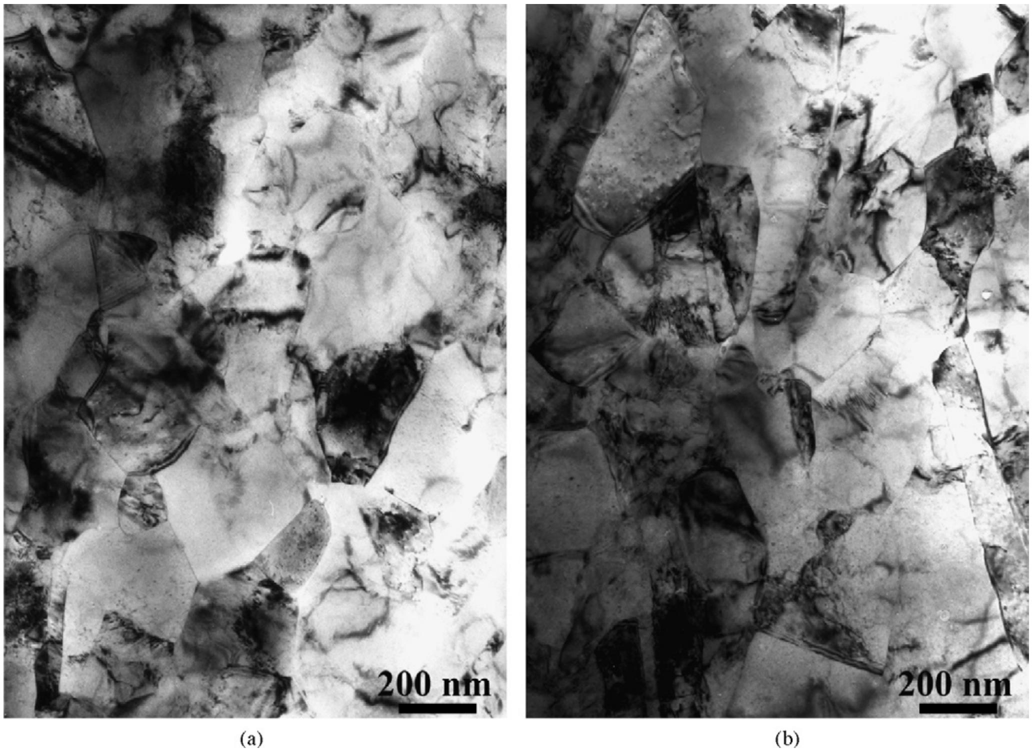

Fig. 1. Transmission electron micrographs of nanostructured copper (a) as-prepared sample and (b) after 40% deformation in compression.

(homologous temperature T/T_m: 0.26–0.29) under ambient atmosphere at strain rates ranging between $1 \times 10^{-5}\,s^{-1}$ and $2 \times 10^{-3}\,s^{-1}$. For $T = 353$ K, the test has been carried out up to $9 \times 10^{-3}\,s^{-1}$. Specimens tested were cylindrical with gage length of 6 mm and diameter of 4.5 mm.

Values of m are calculated at a jump, from strain-rate $\dot{\varepsilon}_1$ at flow stress σ_1 to $\dot{\varepsilon}_2$ at σ_2, by: $m = \ln(\sigma_2/\sigma_1)/\ln(\varepsilon_2/\varepsilon_1)$. In [16], Duhamel et al. have shown also that, as observed in the analysis of superplasticity, values of $\ln \sigma$ and $\ln \dot{\varepsilon}$, are correlated by a sigmoidal function. The fit of the data then allows to calculate values of m as a function of $\dot{\varepsilon}$ (and σ).

3. Validity of the velocity jumps tests

Measurements should be made on different samples at constant strain. This is because the strength of the material is usually dependant on the strain history and the related variations of the microstructure. Validity of the jump-test was evaluated by Yoshizawa et al. [17] who have examined in details the various techniques for the measurements of m and concluded that except for relaxation, values of m are comparable from jump tests and constant crosshead speed tests (within the error range). Cheng et al. [6] found the same m values with the two techniques for nanocrystalline copper (grain size 60 nm) prepared by ball-milling. No substantial change in microstructure was observed after deformation (at least for temperature below 100 °C), from transmission electron microscopy experiments Fig. 1a and b are respectively micrographs of nanocopper before and after 40% deformation in compression. It is not excluded that the material undergoes some slight grain coarsening for $T > 100$ °C. This point is currently examined in details. Finally, it should be noticed that the largest value of m was obtained at the onset of the plastic strain and is provided with an error of about 10%, being larger than the predicted difference between jump test and constant crosshead speed test.

4. Results and discussion

Example of strain-rate curve performed at room temperature, between $1 \times 10^{-5}\,s^{-1}$ and $3 \times 10^{-4}\,s^{-1}$, is presented in Fig. 2. On the plot, similar test carried out on microcrystalline copper (μc-Cu) sample partially work-hardened is reported. The difference between the two materials is clear, with a significant strain-rate sensitivity for nc-Cu whereas the coarse-grained copper yield strength is nearly non-dependent with the strain

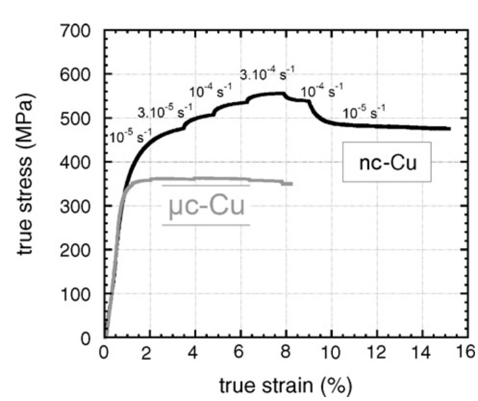

Fig. 2. Comparison between nc-Cu and μc-Cu for room temperature compressive strain rate jump experiments between $10^{-5}\,s^{-1}$ and $3 \times 10^{-4}\,s^{-1}$.

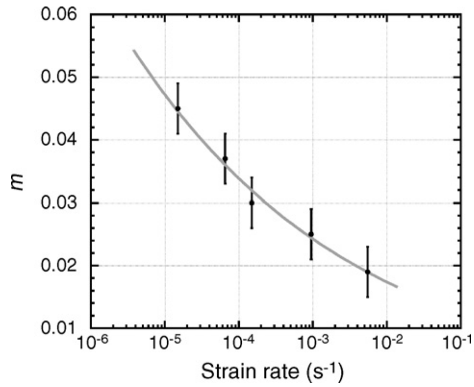

Fig. 3. Strain rate sensitivity m as a function of the logarithm of the strain rate.

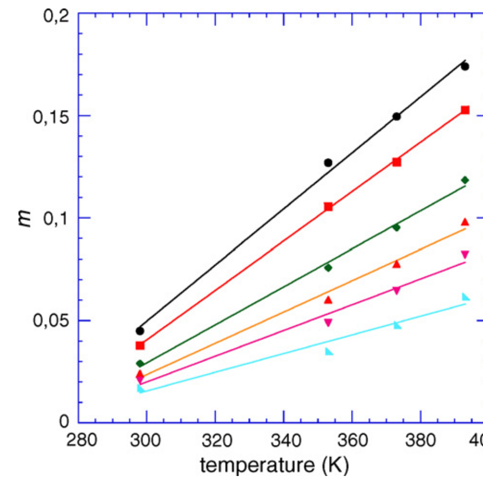

Fig. 4. Strain rate sensitivity m as a function of temperature at $\dot{\varepsilon} = 10^{-5}\,s^{-1}$ (lower slop), $3 \times 10^{-5}\,s^{-1}$, $10^{-4}\,s^{-1}$, $3 \times 10^{-4}\,s^{-1}$, $7 \times 10^{-4}\,s^{-1}$, $2 \times 10^{-3}\,s^{-1}$ (higher slop).

rate with $m = 0.008$. Values of m measured over the range from $1 \times 10^{-5}\,s^{-1}$ to $1.8 \times 10^{-2}\,s^{-1}$ at room temperature are plotted with respect to the logarithm of the strain rate in Fig. 3. Trend is that m decreases with the strain rate. The largest value is $m = 0.045 \pm 0.004$, measured at $1 \times 10^{-5}\,s^{-1}$. Comparison with literature data is relevant if m values are measured in the same conditions; that is within the same range of strain rate and using the same type of measurement technique. Wang et al. [5] reported that for ECAP copper with grain size of 300 nm, $m = 0.015$ at $1 \times 10^{-5}\,s^{-1}$. $m = 0.019$ was also reported for ECAP copper followed by cold rolling [18]. Except residual porosity, this fine grains material is close to our material in terms of microstructure and nature of the grain boundaries. The comparison emphasizes the grain size effect on the rheology of the fine grains metals. In contrast, the work by Lu et al. [7] reveals microstructure effect on the rheology. Electrodeposition is able to produce nanocopper constituted of 500 nm grains divided in 90 nm-sized domains by twin boundaries. $m = 0.025 \pm 0.009$ at $6 \times 10^{-4}\,s^{-1}$ to $6 \times 10^{-1}\,s^{-1}$ is measured by nanoindentation. From Fig. 3, the same value is obtained but at $6 \times 10^{-4}\,s^{-1}$ and our m value drops to 0.02 close to $1 \times 10^{-2}\,s^{-1}$. In fact jump test, featured by control of the strain-rate, seems not directly comparable to nanoindentation, which is more similar to creep test at constant stress. In [19] Langlois reported $m = 0.04 \pm 0.0024$ at $3 \times 10^{-4}\,s^{-1}$ to $1 \times 10^{-3}\,s^{-1}$ by nanoindentation, which is more in the range of the strain rate of Lu et al. experiment and reveals grain-boundaries nature effect on the rheology. Langlois also showed the over estimate of m by nanoindentation, with $m = 0.103 \pm 0.015$ at $1 \times 10^{-5}\,s^{-1}$ to $6 \times 10^{-5}\,s^{-1}$, which is twice the value measured by Jump test.

Our main technical objective is the forming of nanometals as to fabricate micro-objects. Hence, the materials must sustain as large deformation as possible without failure within a reasonable strain rate range; object cannot be shaped at infinitely slow rate. Our nanostructured cooper shows 12% of deformation in tensile test. From Fig. 3, one estimates that $m \approx 0.05$ at $5 \times 10^{-6}\,s^{-1}$ which should lead to about 20% of elongation according to Woodford [10]. In the same way, Woodford predicts about 5% elongation at $10^{-4}\,s^{-1}$, with an estimated $m \approx 0.03$, whilst Langlois obtained tensile elongation of less than 1% [19]. Though elongation at failure is not an ideal measure of ductility, with quite poor reproductibility, in the particular case of powder metallurgy processing, residual porosity is generally the major cause for reducing elongation at failure.

Increasing ductility and shape forming ability of nanometals demands to increase m, which is achievable by increasing temperature. For ultrafine-grained copper (UFG) with grain size 350 nm, produced by severe plastic deformation, $m = 0.11$ at $1 \times 10^{-5}\,s^{-1}$ and at 145 °C [20] (compared to $m = 0.015$ at room temperature [10]). The trend was confirmed as more pronounced for electroplated Ni with grain size of 20 nm [21]. Duhamel has carried out a systematic study of the variation of m with respect to strain rate and temperature on the powder metallurgy nanostructured copper (grain size 90 nm) (Fig. 4). This work is focused on the mechanisms involved in the plasticity of nanometals. In addition relevant data are provided in regards to rheology and forming ability. Materials were tested using jump test, between room temperature and 120 °C for strain rate ranging between $1 \times 10^{-5}\,s^{-1}$ and $2 \times 10^{-3}\,s^{-1}$. The nanocopper undergoes grain coarsening above 140 °C and after 1 h [13]. At constant strain rate, linear correlation is observed between m and T. These investigations show that $m = 0.17$ is obtained at 120 °C and at $1 \times 10^{-5}\,s^{-1}$. According analysis proposed by Woodford [10], our nanostructure copper should undergo about 100% elongation in such conditions, which is much encouraging for forming devices.

5. Conclusion

Nanostructured copper prepared by powder metallurgy, with grain size of about 90 nm, was investigated regarding its thermomechanical behaviour. Strain rate sensitivity was measured by jump tests at room and moderate temperatures. The largest values are $m = 0.045 \pm 0.004$ at $1 \times 10^{-5}\,s^{-1}$, at room temperature and $m = 0.17$ at $1 \times 10^{-5}\,s^{-1}$, at 120 °C just before grain coarsening. These results are encouraging regarding shape forming ability of nanostructured metals, though the best value of m is still far from that for superplastic behaviour.

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